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U. S. DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

METHODS OF ANALYSIS

ADOPTED BY THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, SEPTEMBER 5, 6, AND 7, 1895.

EDITED BY

HARVEY W. WILEY,

SECRETARY.

With the collaboration of

L. L. VAN SLYKE and W. D. BIGELOW

Editorial Committee.

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LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Division of Chemistry,
Washington, D. C., October 22, 1895.

Sir: I transmit herewith for your inspection and approval the manuscript containing the methods of analysis adopted by the Association of Official Agricultural Chemists at its twelfth annual meeting, and ask that it be published as Bulletin No. 46 of the Division of Chemistry of this Department.

Harvey W. Wiley,
Chief of the Division of Chemistry.

Hon. J. Sterling Morton,
Secretary.

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OFFICIAL METHODS OF ANALYSIS ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS AT ITS MEETING, SEPTEMBER 5, 6, AND 7, 1895.[1]

I.—METHODS FOR THE ANALYSIS OF FERTILIZERS.

1. Preparation of Sample.

The sample should be well intermixed, finely ground, and passed through a sieve having circular perforations 1 mm in diameter. The grinding and sifting should be performed as rapidly as possible, to avoid loss or gain of moisture during the operation.

2. Determination of Moisture.

In potash salts, sodium nitrate, and ammonium sulfate heat from 1 to 5 grams at 130° until the weight is constant, and determine the loss in weight as moisture. In all other fertilizers, heat 2 grams, or 5 grams if the sample be very coarse, for five hours, at 100°, in a steam bath.

3. Determination of Phosphoric Acid.

(a) gravimetric method.

(1) *Preparation of reagents.*

(*a*) Ammonium citrate solution.—Dissolve 370 grams of commercial citric acid in 1,500 cc of water; nearly neutralize with commercial ammonia; cool; add ammonia until exactly neutral (testing with saturated alcoholic solution of corallin), and bring to volume of 2 liters. Determine the specific gravity, which should be 1.0900 at 20°, before using.

Optional method.—To 370 grams of commercial citric acid add commercial ammonia, specific gravity 0.96, until nearly neutral; reduce the specific gravity to nearly 1.09 and make exactly neutral, testing as follows: Prepare a solution of fused calcium chlorid, 200 grams to the liter, and add four volumes of strong alcohol. Make the mixture exactly neutral, using a small amount of freshly prepared corallin solution as preliminary indicator, and test finally by withdrawing a portion, diluting with an equal volume of water, and testing with cochineal solution; 50 cc of this solution will precipitate the citric acid from 10 cc of the citrate solution. To 10 cc of the nearly neutral citrate solution add 50 cc of the alcoholic calcium chlorid solution, stir well, filter at once through a folded filter, dilute with an equal volume of water, and test the reaction with neutral solution of cochineal. If acid or alkaline, add ammonia or citric acid, as the case may be, mix, and test again, as before. Repeat this process until a neutral reaction is obtained. Bring to the proper specific gravity of 1.0900 at 20°.

[1] The arrangement of the official methods of analysis is that reported by the editing committee, so modified as to include such changes as were adopted by the association on the recommendation of the various reporters.

(*b*) MOLYBDIC SOLUTION.—Dissolve 100 grams of molybdic acid in 400 grams or 417 cc of ammonia, specific gravity 0.96, and pour the solution thus obtained into 1,500 grams or 1,250 cc of nitric acid, specific gravity 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° deposits no yellow precipitate of ammonium phosphomolybdate. Decant the solution from any sediment and preserve it in glass-stoppered vessels.

(*c*) AMMONIUM NITRATE SOLUTION.—Dissolve 200 grams of commercial ammonium nitrate in water and dilute with water to 2 liters.

(*d*) MAGNESIA MIXTURE.—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding an excess of the latter. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, alumina, and phosphoric acid; filter; add 280 grams of ammonium chlorid, 700 cc of ammonia of specific gravity 0.96, and water enough to make a volume of 2 liters. Instead of the solution of 22 grams of calcined magnesia, 110 grams of crystallized magnesium chlorid ($MgCl_2.6H_2O$) may be used.

(*e*) DILUTE AMMONIA FOR WASHING.—This solution is prepared so as to contain 2.5 per cent NH_3.

(*f*) MAGNESIUM NITRATE SOLUTION.—Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess; boil; filter from the excess of magnesia, ferric oxid, etc., and dilute with water to 2 liters.

(2) *Total phosphoric acid.*

(*a*) METHODS OF MAKING SOLUTION.—Treat 2 grams of the sample by one of the methods given below. After solution, cool, dilute to 200 or 250 cc, mix, and pass through a dry filter.

(a_1) Ignite and dissolve in hydrochloric acid.

(a_2) Evaporate with 5 cc of magnesium nitrate, ignite, and dissolve in hydrochloric acid.

(a_3) Boil with from 20 to 30 cc of strong sulfuric acid, adding from 2 to 4 grams of sodium or potassium nitrate at the beginning of the digestion and a small quantity after the solution has become nearly colorless, or adding the nitrate in small portions from time to time. A Kjeldahl flask marked at 250 cc is recommended. After the solution is colorless, add 150 cc of water and boil for a few minutes, cool, and make up to mark.

(a_4) Digest with strong sulfuric acid and such other reagents as are used in either the plain or modified Kjeldahl or Gunning methods for estimating nitrogen. Do not add any potassium permanganate, but after the solution has become colorless add about 100 cc of water and boil for a few minutes, cool, and make up to a convenient volume; 2.5 grams of substance and a digestion flask marked at 250 cc are recommended.

(a_5) Dissolve in 30 cc of concentrated nitric acid and a small quantity of hydrochloric acid, and boil until organic matter is destroyed.

(a_6) Add 30 cc of concentrated hydrochloric acid, heat, and add cautiously, in small quantities at a time, about 0.5 gram of finely pulverized potassium chlorate, to destroy organic matter.

(a_7) Dissolve in from 15 to 30 cc of strong hydrochloric acid and from 3 to 10 cc of nitric acid. This method is recommended for fertilizers containing much iron or aluminum phosphate.

(*b*) DETERMINATION.—Take an aliquot portion of the solution prepared above, corresponding to 0.25 gram, 0.50 gram, or 1 gram, neutralize with ammonia, and clear with a few drops of nitric acid. In case hydrochloric or sulfuric acid has been used as solvent, add about 15 grams of dry ammonium nitrate or a solution containing that amount. To the hot solution add 50 cc of molybdic solution for every decigram of P_2O_5 that is present. Digest at about 65° for an hour, filter, and wash with cold water, or preferably ammonium nitrate solution. Test the filtrate for phos-

phoric acid by renewed digestion and addition of more molybdic solution. Dissolve the precipitate on the filter with ammonia and hot water and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (about 1 drop per second), stirring vigorously. After fifteen minutes add 30 cc of ammonia solution of density 0.96. Let stand for some time; two hours is usually enough. Filter, wash with 2.5 per cent NH_3 until practically free from chlorids, ignite to whiteness or to a grayish white, and weigh.

(3) *Water-soluble phosphoric acid.*

Place 2 grams on a 9 cm filter, wash with successive small portions of water, allowing each portion to pass through before adding more, until the filtrate measures about 250 cc. (If the filtrate be turbid, add a little nitric acid.) Make up to any convenient definite volume; mix well; take any convenient portion and determine the phosphoric acid as under total phosphoric acid.

(4) *Citrate-insoluble phosphoric acid.*

(*a*) IN ACIDULATED GOODS.—Heat 100 cc of strictly neutral ammonium citrate solution of 1.0900 specific gravity to 65° in a flask placed in a bath of warm water, keeping the flask loosely stoppered to prevent evaporation. When the citrate solution in the flask has reached 65°, drop into it the filter containing the washed residue from the water-soluble phosphoric acid determination, stopper tightly with a smooth rubber stopper, and shake violently until the filter paper is reduced to a pulp. Place the flask again in the bath and maintain the water in the bath at such a temperature that the contents of the flask will stand at exactly 65°. Shake the flask every five minutes. At the expiration of exactly thirty minutes from the time the filter and residue were introduced, remove the flask from the bath and immediately filter as rapidly as possible. Wash thoroughly with water at 65°. Transfer the filter and its contents to a crucible, ignite until all organic matter is destroyed add from 10 to 15 cc of strong hydrochloric acid, and digest until all phosphate is dissolved; or return the filter with contents to the digestion flask, add from 30 to 35 cc strong nitric acid and from 5 to 10 cc strong hydrochloric acid, and boil until all phosphate is dissolved. Dilute the solution to 200 cc. If desired, the filter and its contents can be treated according to methods (a_2), (a_3), or (a_4), under total phosphoric acid. Mix well; filter through a dry filter; take a definite portion of the filtrate and determine phosphoric acid as under total phosphoric acid.

(*b*) IN NONACIDULATED GOODS.—In case a determination of citrate-insoluble phosphoric acid is required in nonacidulated goods it is to be made by treating 2 grams of the phosphatic material, without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, etc.) the residue insoluble in ammonium citrate is to be treated by one of the processes described under total phosphoric acid (a_2), (a_3), or (a_4).

(5) *Citrate-soluble phosphoric acid.*

The sum of the water-soluble and citrate-insoluble subtracted from the total gives the citrate-soluble phosphoric acid.

(b) VOLUMETRIC METHOD.

[This method is not official, but the association directed that it be printed immediately after the official gravimetric method, with the understanding that it be further investigated, and be brought before the association at its next meeting for adoption.]

(1) *Preparation of reagents.*

(*a*) MOLYBDIC SOLUTION.—To 100 cc of molybdic solution, prepared as directed on page 12, add 5 cc of nitric acid (specific gravity, 1.42). This solution should be filtered each time before using.

(*b*) POTASSIUM NITRATE OR AMMONIUM NITRATE SOLUTION FOR WASHING.—Dissolve 3 grams of either salt in 100 cc of water.

(*c*) NITRIC ACID SOLUTION FOR WASHING.—Dilute 100 cc of nitric acid (specific gravity, 1.42) to 1 liter with water.

(*d*) STANDARD POTASSIUM-HYDROXID SOLUTION.—This solution contains 18.17106 grams of potassium hydroxid to the liter. It is prepared by diluting 323.81 cc of normal potassium hydroxid, which has been freed from carbonates by barium hydroxid, to 1 liter. One hundred cc of the solution should neutralize 32.38 cc of normal acid. One cc is equal to 1 mg P_2O_5 (1 per cent P_2O_5 on basis of 0.1 gram of substance).

(*e*) STANDARD NITRIC ACID SOLUTION.—The strength of this solution is the same as, or one-half that of, the standard alkali solution, and is determined by titrating against that solution, using phenolphthalein as indicator.

(*f*) PHENOLPHTHALEIN SOLUTION.—One gram of phenolphthalein is dissolved in 100 cc of alcohol.

(2) *Total phosphoric acid.*

(*a*) METHODS OF MAKING SOLUTION.—Dissolve according to methods (a_2), (a_5), (a_6), or (a_7), (p. 12,) preferably by (a_5), when these acids are a suitable solvent, and dilute to 200 cc with water.

(*b*) DETERMINATION.

(b_1) *In samples containing less than 5 per cent of P_2O_5.*—To 40 cc of the solution prepared as directed above (corresponding to 0.4 gram of the substance), add 10 cc of concentrated nitric acid (5 cc if the solution has been made according to (a_5)), and then add ammonia until a slight permanent precipitate begins to form, and dilute to 60–75 cc. Heat on the water bath to the temperature of 60°–65°, add 50 cc of freshly filtered molybdic solution ((1) (*a*)) for each decigram of P_2O_5 present, and digest at this temperature six minutes from the time the molybdic solution is added (from eight to ten minutes may be necessary if less than 1 per cent of P_2O_5 is present).

Decant the clear liquid on the filter as quickly as possible.[1] Wash the precipitate by decantation twice with the nitric acid solution ((1) (*c*)), using each time 50–75 cc of the solution, agitating thoroughly, and allowing the precipitate to settle completely, once with the same amount of the potassium nitrate or ammonium nitrate solution ((1) (*b*)), and transfer it to the filter and wash it till the filtrate is no longer acid (250 cc of water are usually sufficient). Now wash the precipitate with the filter into the beaker, add an excess of the standard potassium hydroxid solution ((1) (*d*)) and a few drops of the phenolphthalein solution ((1) (*f*)), and titrate the excess of alkali by adding the standard nitric acid solution ((1) (*e*)) until the color disappears.

The number of cubic centimeters of alkali used, less the number of cubic centimeters of acid necessary to neutralize the solution, is equal to the number of milligrams of P_2O_5 present.

(b_2) *In samples containing 5 per cent of P_2O_5 or more.*—To 20 cc of the solution prepared according to one of the methods given under (*a*) page 12 (equivalent to 0.2 gram of the substance), add 10 cc of concentrated nitric acid (5 cc if the solution was prepared according to (a_5)) and then add ammonia until a slight precipitate just begins to form, dilute to about 100 cc, and proceed as under (b_1).

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[1] Filter as quickly as possible (using pressure) on a 3-inch perforated porcelain funnel (Hirsch through a disk of soft filter paper, a Gooch crucible with one or two pieces of filter paper slightly larger than the bottom of the crucible tightly pressed against the bottom, or a 3-inch funnel fitted with a perforated platinum cone or disk covered with coarse asbestus. Filter paper may also be used, but the other filters in the order named are much to be preferred. It is especially recommended that the 3-inch Hirsch funnel be used where possible, as it permits of rapid filtration, and easy and thorough washing.

(3) *Water-soluble phosphoric acid.*

Dissolve according to directions given under (3) page 13. To an aliquot portion of the solution corresponding to 0.2 or 0.4 gram, add 10 cc of concentrated nitric acid and then ammonia until a slight permanent precipitate begins to form, dilute to 60–75 cc, and proceed as under (2) (b_1).

(4) *Citrate-insoluble phosphoric acid.*

Make the solution according to the directions given under (4) page 13 and determine the phosphoric acid in an aliquot portion corresponding to 0.4 gram, as directed in (2) (b_1).

(5) *Citrate-soluble phosphoric acid.*

The sum of the water-soluble and citrate-insoluble subtracted from the total gives the citrate-soluble phosphoric acid.

4. Determination of Nitrogen.

(a) Kjeldahl method.

[Not applicable in the presence of nitrates.]

(1) *Preparation of reagents.*

(*a*) Standard acid solution.

(a_1) *Standard hydrochloric acid,* the absolute strength of which has been determined by precipitating with silver nitrate and weighing the silver chlorid as follows:

To any convenient quantity of the acid to be standardized, add solution of silver nitrate in slight excess, and 2 cc pure nitric acid, specific gravity, 1.2. Heat to boiling point, and keep at this temperature for some minutes without allowing violent ebullition, and with constant stirring, until the precipitate assumes the granular form. Allow to cool somewhat, and then filter through asbestus. Wash the precipitate by decantation, with 200 cc of very hot water, to which has been added 8 cc of nitric acid and 2 cc of dilute solution of silver nitrate containing 1 gram of the salt in 100 cc of water. The washing by decantation is performed by adding the hot mixture in small quantities at a time, beating up the precipitate well with a thin glass rod after each addition. The pump is kept in action all the time; but to keep out dust during the washing, the cover is only removed from the crucible when the fluid is to be added.

Put the capsule and precipitate aside, return the washings once through the asbestus so as to obtain them quite clear, and set aside to recover excess of silver. Rinse the receiver and complete the washing of the precipitate with about 200 cc of cold water. Half of this is used to wash by decantation and the remainder to transfer the precipitate to the crucible. Finish washing in the crucible, the lumps of silver chlorid being broken down with the glass rod. Remove the second filtrate from the receiver and pass about 20 cc of 98 per cent alcohol through the precipitate. Dry at 140° to 150°. Exposure at this temperature for half an hour is found more than sufficient to dry the precipitate thoroughly.

(a_2) *Standard sulfuric acid,* the absolute strength of which has been determined by precipitation with barium chlorid.

For ordinary work half normal acid is recommended, i. e., acid containing 18.2285 grams of hydrochloric acid or 24.5185 grams sulfuric acid to the liter. For work in determining very small amounts of nitrogen, one-tenth normal acid is recommended. In titrating mineral acids against ammonia solutions, use cochineal as indicator.

(*b*) STANDARD ALKALI SOLUTION.—The strength of this solution, relative to the acid, must be accurately determined. One-tenth normal ammonia solution, i. e., containing 1.7051 grams of ammonia to the liter, is recommended for accurate work.

(*c*) SULFURIC ACID.—The sulfuric acid used should have a specific gravity of 1.84 and be free from nitrates, and also from ammonium sulfate, which is sometimes added in the process of manufacture to destroy oxids of nitrogen.

(*d*) METALLIC MERCURY OR MERCURIC OXID.—If mercuric oxid is used, it should be prepared in the wet way, but not from mercuric nitrate.

(*e*) POTASSIUM PERMANGANATE.—This reagent is used in a finely pulverized state.

(*f*) GRANULATED ZINC, PUMICE STONE, OR ZINC DUST.—One of these reagents is added to the contents of the distillation flasks, when found necessary, in order to prevent bumping. When zinc dust is used, 0.5 gram will be found sufficient.

(*g*) POTASSIUM SULFID SOLUTION.—A solution of 40 grams of commercial potassium sulfid in one liter of water.

(*h*) SODIUM HYDROXID SOLUTION.—A saturated solution of sodium hydroxid free from nitrates.

(*i*) INDICATOR.—A solution of cochineal is prepared by digesting and frequently agitating 3 grams of pulverized cochineal in a mixture of 50 cc of strong alcohol and 200 cc of distilled water for a day or two at ordinary temperatures. The filtered solution is employed as indicator.

(2) *Apparatus.*

(*a*) KJELDAHL DIGESTION FLASKS.—These are pear-shaped, round-bottomed flasks, made of hard, moderately thick, well-annealed glass, having a total capacity of about 250 cc. They are 22 cm long, and have a maximum diameter of 6 cm, tapering gradually to a long neck, which is 2 cm in diameter at the narrowest part and flared a little at the edge.

(*b*) DISTILLATION FLASKS.—For distillation, a flask of ordinary shape, of about 550 cc capacity, may be used. It is fitted with a rubber stopper, and with a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation. The bulbs may be about 3 cm in diameter, the tubes being of the same diameter as the condenser and cut off obliquely at the lower end, which is fastened to the condenser by a rubber tube.

(*c*) KJELDAHL FLASKS FOR BOTH DIGESTION AND DISTILLATION.—These are pear-shaped, round-bottomed flasks, having a total capacity of about 550 cc, made of hard, moderately thick, and well-annealed glass. When used for distillation, the flasks are fitted with rubber stoppers and bulb tubes, as given under distillation flasks.

(3) *Determination.*

(*a*) THE DIGESTION.—From 0.7 to 3.5 grams of the substance to be analyzed, according to its proportion of nitrogen, are brought into a digestion flask with approximately 0.7 gram of mercuric oxid, or its equivalent in metallic mercury, and 20 cc of sulfuric acid. The flask is placed in an inclined position, and heated below the boiling point of the acid for from five to fifteen minutes, or until frothing has ceased. If the mixture froths badly, a small piece of paraffin may be added to prevent it. The heat is then raised until the acid boils briskly. No further attention is required till the contents of the flask have become a clear liquid, which is colorless or at least has only a very pale straw color. The flask is then removed from the flame, held upright, and, while still hot, potassium permanganate is dropped in carefully and in small quantities at a time, till, after shaking, the liquid remains of a green or purple color.

(*b*) THE DISTILLATION.—After cooling, the contents of the flask are transferred to the distilling flask with about 200 cc of water, with a few pieces of granulated zinc, pumice stone, or 0.5 gram of zinc dust when found necessary to keep the contents of the flask from bumping, and 25 cc of potassium sulfid solution are added, shaking

the flask to mix its contents. Next add 50 cc of the soda solution, or sufficient to make the reaction strongly alkaline, pouring it down the side of the flask so that it does not mix at once with the acid solution. Connect the flask with the condenser, mix the contents by shaking, and distill until all ammonia has passed over into the standard acid. The first 150 cc of the distillate will generally contain all the ammonia. This operation usually requires from forty minutes to one hour and a half. The distillate is then titrated with standard alkali.

The use of mercuric oxid in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in case of substances most difficult to oxidize, and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. The potassium sulfid removes all the mercury from the solution, and so prevents the formation of mercur-ammonium compounds which are not completely decomposed by the sodium hydroxid. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the reagents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present, which might otherwise escape notice.

(b) GUNNING METHOD.

[Not applicable in the presence of nitrates.]

(1) *Preparation of reagents.*

(*a*) POTASSIUM SULFATE.—This reagent should be pulverized before using.

(*b*) SULFURIC ACID.—The same sulfuric acid is used as in the Kjeldahl method ((*c*), p. 16).

(2) *Apparatus.*

The apparatus used is the same as that described in the Kjeldahl method (p. 16).

(3) *Determination.*

In a digestion flask holding from 250 to 500 cc, place from 0.7 to 3.5 grams of the substance to be analyzed, according to its proportion of nitrogen. Then add 10 grams of powdered potassium sulfate and from 15 to 25 cc (ordinarily about 20 cc) of concentrated sulfuric acid. Conduct the digestion as in the Kjeldahl process, starting with a temperature below boiling point and increasing the heat gradually until frothing ceases. Digest until colorless or nearly so. Do not add either potassium permanganate or potassium sulfid. Dilute, neutralize, and distill as in the Kjeldahl method. In neutralizing, it is convenient to add a few drops of phenolphthalein indicator, by which one can tell when the acid is completely neutralized, remembering that the pink color, which indicates an alkaline reaction, is destroyed by a considerable excess of strong fixed alkali. The distillation and titration are conducted as in the Kjeldahl method.

(c) KJELDAHL METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.

(1) *Preparation of reagents.*

Besides the reagents given under the Kjeldahl method, there will be needed—

(*a*) ZINC DUST.—This should be an impalpable powder; granulated zinc or zinc filings will not answer.

(*b*) SODIUM THIOSULFATE.

(*c*) COMMERCIAL SALICYLIC ACID.

(2) *Apparatus.*

The apparatus used is the same as in the Kjeldahl method ((a), (2)).

(3) *Determination.*

Bring from 0.7 to 3.5 grams of the substance to be analyzed into a Kjeldahl digestion flask, add 30 cc of sulfuric acid containing 1 gram of salicylic acid and shake until thoroughly mixed with the acid, then add 5 grams of crystallized sodium thiosulfate; or add to the substance 30 cc of sulfuric acid containing 2 grams of salicylic acid, then add gradually 2 grams of zinc dust, shaking the contents of the flask at the same time. Finally, place the flask on the stand for holding the digestion flasks, where it is heated over a low flame until all danger from frothing has passed. The heat is then raised until the acid boils briskly and the boiling continued until white fumes no longer pour out of the flask. This requires about five or ten minutes. Add approximately 0.7 gram of mercuric oxid or its equivalent in metallic mercury, and continue the boiling until the liquid in the flask is colorless or nearly so. In case the contents of the flask are likely to become solid before this point is reached, add 10 cc more of sulfuric acid. Complete the oxidation with a little potassium permanganate in the usual way and proceed with the distillation as described in the Kjeldahl method. The reagents should be tested by blank experiments.

(d) GUNNING METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.

(1) *Preparation of reagents.*

Besides the reagents given under the Gunning method, there will be needed—

(*a*) SODIUM THIOSULFATE.

(*b*) COMMERCIAL SALICYLIC ACID.

(2) *Apparatus.*

The apparatus used is the same as that given under the Kjeldahl method ((a), (2)).

(3) *Determination.*

In a digestion flask holding from 250 to 500 cc place from 0.7 to 3.5 grams of the substance to be analyzed, according to the amount of nitrogen present. Add from 30 to 35 cc of salicylic acid mixture, namely, 30 cc sulfuric acid to 1 gram of salicylic acid, shake until thoroughly mixed, and allow to stand from five to ten minutes, with frequent shaking; then add 5 grams of sodium thiosulfate and 10 grams of potassium sulfate. Heat very gently until frothing ceases, then heat strongly until nearly colorless. Dilute, neutralize, and distill as in the Gunning method.

(e) ABSOLUTE OR CUPRIC OXID METHOD.

[Applicable to all nitrogen determinations.]

(1) *Preparation of reagents.*

(*a*) *Coarse or wire form cupric oxid.*—To be ignited and cooled before using.

(*b*) *Fine cupric oxid.*—Prepared by pounding ordinary cupric oxid in a mortar.

(*c*) *Metallic copper.*—Granulated copper, or fine copper gauze, reduced and cooled in a current of hydrogen.

(*d*) *Sodium bicarbonate.*—Free from organic matter.

(*e*) *Caustic potash solution.*—Make a supersaturated solution of caustic potash in hot water. When the absorption of carbon dioxid ceases to be prompt, the solution must be discarded.

(2) *Apparatus.*

(*a*) *Combustion tube* of best hard Bohemian glass, about 66 cm long and 12.7 mm internal diameter.

(*b*) *Azotometer* of at least 100 cc capacity, accurately calibrated.

(*c*) *Sprengel mercury air pump.*

(*d*) *Small paper scoop,* easily made from stiff writing paper.

(3) *Determination.*

Of ordinary commercial fertilizers use from 1 to 2 grams for analysis. In the case of highly nitrogenized substances the amount to be used must be regulated by the amount of nitrogen estimated to be present. Fill the tube as follows: (1) About 5 cm of coarse cupric oxid. (2) Place on the small paper scoop enough of the fine cupric oxid to fill, after having been mixed with the substance to be analyzed, about 10 cm of the tube; pour on this the substance, rinsing the watch glass with a little of the fine oxid, and mix thoroughly with a spatula; pour into the tube, rinsing the scoop with a little fine oxid. (3) About 30 cm of coarse cupric oxid. (4) About 7 cm of metallic copper. (5) About 6 cm of coarse cupric oxid (anterior layer). (6) A small plug of asbestus. (7) From 0.8 to 1 gram of sodium bicarbonate. (8) A large, loose plug of asbestus. Place the tube in the furnace, leaving about 2.5 cm of it projecting; connect with the pump by a rubber stopper smeared with glycerol, taking care to make the connection perfectly tight.

Exhaust the air from the tube by means of the pump. When a vacuum has been obtained, allow the flow of mercury to continue; light the gas under that part of the tube containing the metallic copper, the anterior layer of cupric oxid (see (5) above), and the sodium bicarbonate. As soon as the vacuum is destroyed and the apparatus filled with carbon dioxid, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer just below the surface of the mercury seal, so that the escaping bubbles will pass into the air and not into the tube, thus avoiding the useless saturation of the caustic potash solution.

When the flow of carbon dioxid has very nearly or completely ceased, pass the delivery tube down into the receiving arm, so that the bubbles will escape into the azotometer. Light the gas under the 30 cm layer of oxid, heat gently for a few moments to drive out any moisture that may be present, and bring to a red heat. Heat gradually the mixture of substance and oxid, lighting one jet at a time. Avoid a too rapid evolution of bubbles, which should be allowed to escape at the rate of about one per second, or a little faster.

When the jets under the mixture have all been turned on, light the gas under the layer of oxid at the end of the tube. When the evolution of gas has ceased, turn out all the lights except those under the metallic copper and anterior layer of oxid, and allow to cool for a few moments. Exhaust with the pump and remove the azotometer before the flow of mercury is stopped. Break the connection of the tube with the pump, stop the flow of mercury, and extinguish the lights. Allow the azotometer to stand for at least an hour, or cool with a stream of water until a permanent volume and temperature have been reached.

Adjust accurately the level of the potassium hydroxid solution in the bulb to that in the azotometer; note the volume of gas, temperature, and height of barometer; make calculation as usual, or read results from tables.

(f) RUFFLE METHOD.

(1) *Preparation of reagents.*

(*a*) Standard solutions and indicator the same as for the Kjeldahl method (p. 15).

(*b*) A mixture of equal parts by weight of fine slaked lime and finely powdered sodium thiosulfate dried at 100°.

(*c*) A mixture of equal parts by weight of finely powdered granulated sugar and flowers of sulfur.

(*d*) Granulated soda-lime, as described under the soda-lime method (p. 20).

(2) *Apparatus.*

(*a*) Combustion tubes of hard Bohemian glass, 70 cm long and 1.3 cm in diameter.

(*b*) Bulbed U-tubes or Will's bulbs, as described under the soda-lime method.

(3) *Determination.*

Clean the U-tube and introduce 10 cc of standard acid.

Fill the tube as follows: (1) A loosely fitting plug of asbestus, previously ignited, and then from 2.5 to 3.5 cm of the thiosulfate mixture. (2) The weighed portion of the substance to be analyzed is intimately mixed with from 5 to 10 grams of the sugar and sulfur mixture. (3) Pour on a piece of glazed paper or in a porcelain mortar a sufficient quantity of thiosulfate mixture to fill about 25 cm of tube; then add the substance to be analyzed, as previously prepared, mix carefully, and pour into the tube; shake down the contents of the tube; rinse off the paper or mortar with a small quantity of the thiosulfate mixture; then fill up with soda-lime to within 5 cm of the end. (4) Place another plug of ignited asbestus at the end of the tube and close with a cork. (5) Hold the tube in a horizontal position and tap on the table until there is a gas channel all along the top. Make connections with the U-tube containing the acid; aspirate and see that the apparatus is tight.

The combustion.—Place the prepared combustion tube in the furnace, letting the open end project a little, so as not to burn the cork. Commence by heating the soda-lime portion until it is brought to a full red heat. Then turn on slowly jet after jet toward the outer end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and the evolution of the gas has ceased and the liquid in the U-tube begins to recede toward the furnace, attach the aspirator to the other limb of the U-tube, break off the end of the combustion tube, and draw a current of air through for a few minutes. Detach the U-tube and wash its contents into a beaker or porcelain dish, add a few drops of the cochineal solution, and titrate.

(g) SODA-LIME METHOD.

[Not applicable in the presence of nitrates.]

(1) *Preparation of reagents.*

(*a*) STANDARD SOLUTIONS AND INDICATOR.—Those mentioned under the Kjeldahl method ((a), (b), and (i), pp. 15–26) may be used.

(*b*) SODA-LIME.—Excellent soda-lime may be easily and quickly prepared by adding 2.5 parts of quicklime to 1 part by weight of commercial caustic soda (such soda as is used in the Kjeldahl method) dissolved in a sufficient amount of water to slake the lime. The mixture is then dried and heated in an iron pot to incipient fusion, and when cold is ground and sifted. Two sizes of granules are required in this method—

(b_1) Fine enough to pass through a 2.5 mm sieve, and thoroughly dry.

(b_2) Fine enough to pass through a 1.25 mm sieve, also thoroughly dry.

(*c*) SODIUM CARBONATE AND LIME OR SLAKED LIME.—Instead of soda-lime, Johnson's mixture of sodium carbonate and lime or slaked lime may be used.

Slaked lime may be granulated by mixing it with a little water to form a thick mass, which is dried in the water oven until hard and brittle. It is then ground and sifted as above. Slaked lime is much easier to work with than soda-lime and gives excellent results, though it is probable that more of it should be used in proportion to the substance to be analyzed than is the case with soda-lime.

(2) *Apparatus.*

(*a*) ASBESTUS.—The asbestus used should be ignited and kept in a glass-stoppered bottle.

(*b*) COMBUSTION TUBES.—These are about 40 cm long and of 12 mm internal diameter, drawn out to a point and closed at one end.

(*c*) U-TUBES.—Large-bulbed U-tubes with glass stopcocks, or Will's tubes with four bulbs.

(3) *Determination.*

The substance to be analyzed should be powdered finely enough to pass through a sieve of 1 mm mesh; 0.7 or 1.4 grams, according to the amount of nitrogen present, are taken for the determination. Into the closed end of the combustion tube put a small loose plug of asbestus, and upon it about 4 cm of fine soda-lime. In a porcelain dish or mortar mix the substance to be analyzed, thoroughly but quickly, with enough fine soda-lime to fill about 16 cm of the tube, or about 40 times as much soda-lime as substance, and put the mixture into the combustion tube as quickly as possible by means of a wide-necked funnel, rinsing out the dish and funnel with a little more fine soda-lime, which is to be put in on top of the mixture. Fill the rest of the tube to about 5 cm of the end with granulated soda-lime, making it as compact as possible by tapping the tube gently while held in a nearly upright position during the filling. The layer of granulated soda-lime should not be less than 12 cm long. Lastly, put in a plug of asbestus about 2 cm long, pressed rather tightly, and wipe out the end of the tube to free it from adhering particles.

Connect the tube by means of a well-fitting rubber stopper or cork with the U-tube or Will's bulbs, containing 10 cc of standard acid, and adjust it in the combustion furnace so that the end projects about 4 cm from the furnace, supporting the U-tube or Will's bulbs suitably. Heat the portion of the tube containing the granulated soda-lime to a moderate redness, and when this is attained extend the heat gradually through the portion containing the substance, so as to keep up a moderate and regular flow of gases through the bulbs, maintaining the heat of the first part until the whole tube is heated uniformly to the same degree. Keep up the heat until gases have ceased bubbling through the acids in the bulbs, and the mixture of substance and soda-lime has become white, or nearly so, which shows that the combustion is finished. The combustion should occupy about three-quarters of an hour, or not more than one hour. Remove the heat, and when the tube has cooled below redness break off the closed tip and aspirate air slowly through the apparatus for two or three minutes, to bring all the ammonia into the acid. Disconnect, wash the acid into a beaker or flask, and titrate with the standard alkali.

During the combustion the end of the tube projecting from the furnace must be kept heated sufficiently to prevent the condensation of moisture, yet not enough to char the stopper. The heat may be regulated by a shield of tin slipped over the projecting end of the combustion tube.

It is found very advantageous to attach a Bunsen valve to the exit tube, allowing the evolved gases to pass out freely, but preventing a violent "sucking back" in case of a sudden condensation of steam in the bulbs.

(h) MAGNESIUM OXID METHOD.

[Applicable only to the determination of ammonia.]

From 0.7 to 3.5 grams of the substance to be analyzed, according to the proportion of ammonia present, are brought into a distillation flask ((2), (*c*), p. 16) with about 200 cc of water and a large excess of magnesia. The flask is then connected with a condenser, and 100 cc of the liquid distilled into standard acid. The residual acid is titrated as in the Kjeldahl method, page 16.

(i) ULSCH METHOD, MODIFIED BY STREET.[1]

[Applicable to all nitric and ammoniacal nitrogen determinations.]

Place 1 gram of the sample in a half liter flat-bottomed flask. Add about 30 cc of water, 1 gram of reduced iron, and 10 cc of a mixture of strong sulfuric acid with an equal volume of water; shake well, and allow to stand for a short time. This

[1]Methods (i) and (j) are not official, but the association directed that they be printed immediately after the official methods, with the understanding that they be further investigated, and be brought before the association at its next meeting for adoption.

will remove the danger of an explosion caused by the otherwise violent action which takes place. Close the neck of the flask with a rubber stopper through which passes a dropping bulb filled with water. The flask having thus been stoppered, place it on a slab to which a moderate heat is applied. Heat the solution slowly, boil it for five minutes, and cool it. Add about 100 cc of water, a little paraffin, and from 7 to 10 grams of magnesium oxid. The flask is then connected with a condenser, such as is used in the Kjeldahl method, and boiled for forty minutes, the ammonia being collected in a known amount of standard acid. When magnesia is used, assurance must be had that it is strongly in excess, and forty minutes are necessary for the complete distillation of the ammonia. The contents of the receiver are titrated as in the Kjeldahl method, page 16.

(j) ZINC-IRON METHOD.

[Applicable to the determination of nitric and ammoniacal nitrogen determinations.]

Ten grams of the sample are dissolved in 500 cc of water. Of this solution 25 cc, corresponding to one-half gram, are placed in a distillation flask of about 400 cc capacity, 120 cc of water added, and also about 5 grams of well washed and dried zinc dust and an equal weight of reduced iron. To the solution are added 80 cc of sodium hydrate of 32° B. The flask is then connected with the condensing apparatus and the distillation carried on synchronously with the reduction, the ammonia being collected in carefully standardized acid. The distillation is continued for one or two hours, or until 100 cc have been distilled. The resulting distillate is titrated as in the Kjeldahl method, page 16.

5. Determination of Potash.

(a) LINDO-GLADDING METHOD.

(1) *Preparation of reagents.*

(*a*) AMMONIUM CHLORID SOLUTION.—Dissolve 100 grams of ammonium chlorid in 500 cc of water, add from 5 to 10 grams of pulverized potassium platinic chlorid, and shake at intervals for six or eight hours. The mixture is then allowed to settle overnight and filtered, and the residue is ready for the preparation of a fresh supply when needed.

(*b*) PLATINUM SOLUTION.—The platinum solution used contains 1 gram of metallic platinum (2.1 grams of H_2PtCl_6) in every 10 cc.

(2) *Methods of making solution.*

(*a*) WITH POTASH SALTS AND MIXED FERTILIZERS.—Boil 10 grams of the sample with 300 cc of water thirty minutes. Except in case of muriate of potash, add to the hot solution a slight excess of ammonia and then a sufficient amount of ammonium oxalate to precipitate all the lime present. The solution is then cooled, diluted to 500 cc, thoroughly mixed, and filtered through a dry filter. In case of muriate of potash the solution is diluted to 500 cc without the addition of ammonia and ammonium oxalate.

(*b*) WITH ORGANIC COMPOUNDS.—In the case of fertilizers supposed to contain organic compounds, like tobacco stems, cotton-seed hulls, etc., saturate 10 grams with strong sulfuric acid, and ignite in a muffle at a low red heat to destroy organic matter. Add a little strong hydrochloric acid and warm slightly in order to loosen the mass from the dish, and proceed as directed under (1).

(3) *Determination.*

(*a*) IN MIXED FERTILIZERS.—Evaporate 50 cc of the solution made according to (2), corresponding to 1 gram of the sample, nearly to dryness, add 1 cc of dilute sulfuric acid (1 to 1), evaporate to dryness and ignite to whiteness. As all the potash

is in form of sulfate, no loss need be apprehended by volatilization of potash, and a full red heat must be maintained until the residue is perfectly white. This residue is dissolved in hot water acidified with a few drops of hydrochloric acid, and so diluted that the platinum chlorid solution which is now added will cause no immediate precipitation. This solution is then evaporated to a thick paste in a small dish, and 80 per cent alcohol (specific gravity, 0.8645) added. In evaporating, special precaution should be taken to prevent absorption of ammonia. The precipitate is washed thoroughly with alcohol by decantation and on the filter, as usual. The washing should be continued even after the filtrate is colorless. Ten cubic centimeters of the ammonium chlorid solution ((1), (*a*)) are now run through the filter, or the washing may be performed in the dish. The 10 cc will contain the bulk of the impurities, and are thrown away. Five or six portions of the ammonium chlorid solution of 10 cc each are now run through the filter. The filter is then washed thoroughly with 80 per cent alcohol (specific gravity, 0.8645), dried, and weighed as usual. Care should be taken that the precipitate is perfectly soluble in water.

(*b*) In muriate of potash.—In the analysis of this salt an aliquot portion of the solution prepared according to (2), containing 0.5 gram of the sample so diluted that on the addition of a slight excess of platinum chlorid no immediate precipitate will result, is evaporated with 10 cc of the platinum solution and a few drops of hydrochloric acid, and washed as directed under (*a*).

(*c*) In sulfate of potash, kainit, etc.—To 25 cc of the solution prepared according to (2), corresponding to 0.5 gram of the sample, add 1 cc of dilute sulfuric acid (1–1), evaporate to dryness and ignite to whiteness, dissolve the residue in water and a few drops of hydrochloric acid, and add 15 cc of the platinum solution. In the analysis of high-grade sulfate and of double-manure salt (potassium sulfate and magnesium sulfate, containing about 27 per cent of potassium oxid), make up the solution as above, but omit the precipitation, evaporation, etc., so dilute an aliquot part equal to 0.5 gram of the substance that on the addition of platinum chlorid no immediate precipitate will result, and add 15 cc of platinum chlorid solution. Then proceed with the evaporation, digestion, etc., as under (*a*). In all cases special care must be taken in the washing with alcohol to remove all the double platinum-sodium chlorid if present. The washing should be continued some time after the filtrate is colorless. Run several portions of the ammonium chlorid solution, of 25 cc each, through the filter, to remove all sulfates and chlorids. Wash finally with 80 per cent alcohol (specific gravity, 0.8645), and dry and weigh as usual.

(b) optional method.

(1) *Preparation of reagent.*

Platinum solution.—The platinum solution used is the same as that described under the Lindo-Gladding method, page 22.

(2) *Method of making solution.*

The solution is made in all cases as directed under the Lindo-Gladding method, page 22.

(3) *Determination.*

Dilute 25 cc of the solution made as directed under (2) (50 cc, if less than 10 per cent of potassium oxid is present) to 150 cc, heat to 100°, and add, drop by drop with constant stirring, a slight excess of barium chlorid. Without filtering, in the same manner add barium hydrate in slight excess. Filter while hot and wash until the precipitate is free from chlorids. Add to the filtrate 1 cc of strong ammonium hydrate, and then a saturated solution of ammonium carbonate until the excess of barium is precipitated. Heat and add, in fine powder, 0.5 gram of pure oxalic acid or 0.75 gram of ammonium oxalate. Filter and wash free from chlorids, evaporate the

filtrate to dryness in a platinum dish, and ignite carefully over the free flame below a red heat until all volatile matter is driven off.

The residue is digested with hot water, filtered through a small filter, and the filtrate so diluted that on the addition of a slight excess of the platinum chlorid solution no immediate precipitate will result. It is acidified with a few drops of hydrochloric acid in a porcelain dish, and from 5 to 10 cc of the platinum chlorid solution are added. The mixture is evaporated on a water bath to a thick sirup, as above, treated with 80 per cent alcohol, washed by decantation, collected in a Gooch crucible or other form of filter, washed with 80 per cent alcohol (specific gravity, 0.8645), dried for 30 minutes at 100°, and weighed.

It is desirable, if there be an appearance of foreign matter in the double salt, that it should be washed, according to the previous method, with several portions of the ammonium chlorid solution of 10 cc each.

(c) FACTORS.

The use of the factors 0.3056 for converting potassium-platinum chlorid to potassium chlorid and 0.19308 for converting to potassium oxid is continued.

II.—METHODS FOR THE ANALYSIS OF FOODS.

1. Preparation of Sample.

The substance is to be ground and passed through a sieve with circular holes 1 mm in diameter.

2. Determination of Moisture.

Dry from 2 to 3 grams of the substance for five hours, at the temperature of boiling water, in a current of dry hydrogen. If the substance be held in a glass vessel, the latter should not be in contact with the boiling water.

3. Determination of Ash.

Char from 2 to 3 grams of the substance and burn to whiteness at the lowest possible red heat. If a white ash can not be obtained in this manner, exhaust the charred mass with water; collect the insoluble residue on a filter, burn, add this ash to the residue from the evaporation of the aqueous extract, and heat the whole to a low redness till the ash is white.

4. Determination of Ether Extract.

(a) PREPARATION OF ANHYDROUS ETHER.

To prepare the anhydrous alcohol-free ether required for the estimation of fat, wash any of the commercial brands of ether with two or three successive portions of distilled water, and add solid caustic soda or potash until most of the water has been abstracted from the ether. Carefully cleaned metallic sodium, cut into small pieces, is added until there is no further evolution of hydrogen gas. The ether thus dehydrated must be kept over metallic sodium, and should be only lightly stoppered in order to allow any accumulating hydrogen gas to escape. It may be drawn off with a pipette as required.

(b) DETERMINATION.

(1) *Direct method.*

Extract from 2 to 3 grams of the substance dried as for the determination of the moisture, with anhydrous alcohol-free ether, sixteen hours. Dry the extract, by exposure at the temperature of boiling water, to constant weight.

(2) *Indirect method.*

In determining hygroscopic water, as above, continue the drying until the loss of weight in thirty minutes is reduced to 1 mg or less, extract the dried substance for sixteen hours as directed, dry again, and give loss of weight as ether extract.

5. Determination of Crude Protein.

Determine nitrogen as directed for nitrogen in fertilizers and multiply the result by 6.25 for the crude protein.

6. Determination of Albuminoid Nitrogen by Stutzer's Method.

(a) preparation of reagent.

Prepare cupric hydrate as follows: Dissolve 100 grams of pure cupric sulfate in 5 liters of water, and add 25 cc of glycerol, and then a dilute solution of sodium hydrate until the liquid is alkaline; filter; rub the precipitate up with water containing 5 cc of glycerol per liter, and wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent of glycerol, thus preparing a uniform gelatinous mass that can be measured out with a pipette. Determine the quantity of cupric hydrate per cubic centimeter of this mixture.

(b) determination.

To 0.7 gram of the substance add 100 cc of water in a beaker, heat to boiling, or, in the case of substances rich in starch, heat on the water bath ten minutes; add a quantity of cupric hydrate mixture containing about 0.5 gram of the hydrate; stir thoroughly, filter when cold, wash with cold water, and, without removing the precipitate from the filter determine nitrogen according to one of the methods given for the determination of nitrogen in fertilizers (p. 15), adding sufficient potassium sulfid solution to completely precipitate all copper and mercury. The filter papers used must be practically free from nitrogen. If the substance examined consists of seed of any kind, or residues of seeds, such as oil cake or anything else rich in alkaline phosphates, add a few cubic centimeters of a concentrated solution of alum just before adding the cupric hydrate, and mix well by stirring. This serves to decompose the alkaline phosphates. If this be not done, cupric phosphate and free alkali may be formed, and the protein copper may be partially dissolved in the alkaline liquid.

7. Determination of Crude Fiber and Carbohydrates.

For these methods, see under Determination of Carbohydrates in Agricultural Products, VI, 7.

III.—METHODS FOR THE ANALYSIS OF DAIRY PRODUCTS.

1. Butter Analysis.

(a) preparation of sample.

If large quantities of butter are to be sampled, a butter trier or sampler may be used. The portions thus drawn, about 500 grams, are to be perfectly melted in a closed vessel at as low a temperature as possible, and when melted the whole is to be shaken violently for some minutes till the mass is homogeneous, and sufficiently solidified to prevent the separation of the water and fat. A portion is then poured into the vessel from which it is to be weighed for analysis, and should nearly or quite fill it. This sample should be kept in a cold place till analyzed.

(b) DETERMINATION OF WATER.

From 1.5 to 2.5 grams are dried to constant weight at the temperature of boiling water in a dish with flat bottom, having a surface of at least 20 square centimeters.

The use of clean dry sand or asbestus with the butter is admissible, and is necessary if a dish with round bottom is employed.

(c) DETERMINATION OF ETHER EXTRACT.

(1) *Direct method.*

The dry butter from the water determination is dissolved in the dish with absolute ether, or with 76° petroleum ether. The contents of the dish are then transferred to a weighed Gooch crucible with the aid of a wash bottle filled with the solvent, and are washed till free from fat. The crucible and contents are heated at the temperature of boiling water till the weight is constant. The weight of fat is calculated from the data obtained.

(2) *Indirect method.*

Water may be determined by drying the butter on asbestus or sand, and the fat extracted by anhydrous alcohol-free ether. The extract, after evaporation of the ether, is heated to constant weight at the temperature of boiling water and weighed.

(d) DETERMINATION OF CASEIN, ASH, AND CHLORIN.

The crucible containing the residue from the fat determination is covered and heated, gently at first, gradually raising the temperature to just below redness. The cover may then be removed and the heat continued till the contents of the crucible are white. The loss in weight of the crucible and contents represents casein, and the residue in the crucible mineral matter. In this mineral matter, dissolved in water slightly acidulated with nitric acid, chlorin may be determined gravimetrically with silver nitrate, or volumetrically, using potassium chromate as an indicator.

(e) DETERMINATION OF SALT.

Weigh in a counterpoised beaker from 5 to 10 grams of the butter. The butter is placed, in portions of about 1 gram at a time, in the beaker, these portions being taken from different parts of the sample. Hot water is now added (about 20 cc) to the beaker containing the butter, and after it has melted the liquid is poured into the bulb of a separating funnel. The stopper is now inserted and the contents shaken for a few moments. After standing until the fat has all collected on top of the water, the stopcock is opened and the water is allowed to run into an Erlenmeyer flask, being careful to let none of the fat globules pass. Hot water is again added to the beaker, and the extraction is repeated from 10 to 15 times, using each time from 10 to 20 cc of water. The resulting washings contain all but a mere trace of the sodium chlorid originally present in the butter. The sodium chlorid is now determined in the filtrate by a standard solution of silver nitrate, using a few drops of a solution of potassium chromate as an indicator.

(f) DETERMINATION OF VOLATILE ACIDS.

(1) *Preparation of reagents.*

(*a*) CAUSTIC SODA SOLUTION.—One hundred grams of sodium hydroxid are dissolved in 100 cc of distilled water. The caustic soda should be as free as possible from carbonates, and be preserved out of contact with the air.

(*b*) Caustic potash solution.—Dissolve 100 grams of the purest potassium hydroxid in 58 grams of hot distilled water. Allow to cool in a stoppered vessel, decant the clear solution, and preserve in a vessel out of contact with the air.

(*c*) Alcohol, of about 95 per cent, redistilled with caustic soda.

(*d*) Acid.—Solution of sulfuric acid containing 25 cc of strongest sulfuric acid in 1,000 cc of water.

(*e*) Barium hydroxid.—An accurately standardized, approximately decinormal solution of barium hydroxid.

(*f*) Indicator.—Dissolve 1 gram of phenolphthalein in 100 cc of alcohol.

(2) *Apparatus.*

(*a*) Saponification flasks, of from 250 to 300 cc capacity, of hard, well-annealed glass, capable of resisting the tension of alcohol vapor at 100°.

(*b*) A pipette graduated to deliver 40 cc.

(*c*) Distilling apparatus.

(*d*) Burette.—An accurately calibrated burette reading to tenths of a cubic centimeter.

(3) *Determination.*

(*a*) Weighing the fat.—The butter or fat to be examined should be melted and kept in a dry, warm place at about 60° for two or three hours, until the water and curd have entirely settled out. The clear supernatant fat is poured off and filtered through a dry filter paper in a jacketed funnel containing boiling water. Should the filtered fat, in a fused state, not be perfectly clear, it must be filtered a second time.

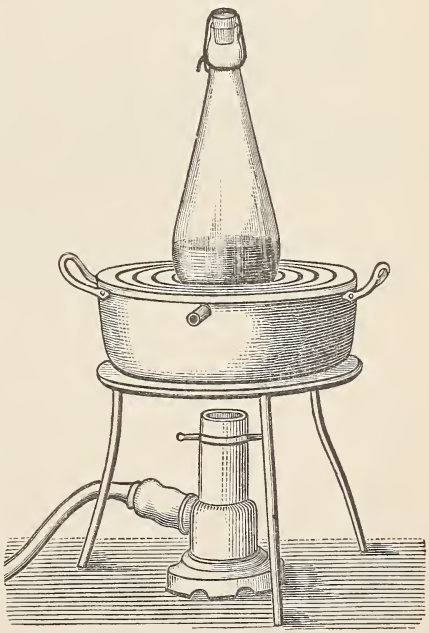

Fig. 1.—Saponification flask.

The saponification flasks are prepared by thoroughly washing with water, alcohol, and ether, wiping perfectly dry on the outside, and heating for one hour at the temperature of boiling water. The flasks should then be placed in a tray by the side of the balance and covered with a silk handkerchief until they are perfectly cool. They must not be wiped with a silk handkerchief within fifteen or twenty minutes of the time they are weighed. The weight of the flasks having been accurately determined, they are charged with the melted fat in the following way:

A pipette with a long stem, marked to deliver 5.75 cc, is warmed to a temperature of about 50°. The fat, having been poured back and forth once or twice into a dry beaker in order to thoroughly mix it, is taken up in the pipette and the nozzle of the pipette carried to near the bottom of the flask, having been previously wiped to remove any adhering fat, and 5.75 cc of fat are allowed to flow into the flask. After the flasks have been charged in this way they should be re-covered with the silk handkerchief and allowed to stand fifteen or twenty minutes, when they are again weighed.

(*b*) SAPONIFICATION.

(b_1) *In the presence of alcohol.*—Ten cc of 95 per cent alcohol are added to the fat in the flask, and then 2 cc of the caustic soda solution. A soft cork stopper is now inserted in the flask and tied down with a piece of twine. The saponification is then completed by placing the flask upon the water or steam bath. The flask during the saponification, which should last one hour, should be gently rotated from time to time, being careful not to project the soap for any distance up its sides. At the end of an hour the flask, after having been cooled to near the room temperature, is opened.

(b_2) *Optional method—without the use of alcohol.*—To avoid the danger of loss from the formation of ethers, and the trouble of removing the alcohol after saponification, the fat may be saponified with a solution of caustic potash in a closed flask without using alcohol. The operation is carried on exactly as indicated above for saponification in the presence of alcohol, using caustic potash solution instead of caustic soda, and omitting the operation for volatilizing the alcohol. For the saponification, use 2 cc of the caustic potash solution, which are poured on the fat after it has solidified in the flask. Great care must be taken that none of the fat be allowed to rise on the sides of the saponifying flask to a point where it can not be reached by the alkali. During the process of saponification the flask can only be very gently rotated in order to avoid the difficulty mentioned. This process is not recommended with any apparatus except a closed flask with round bottom. Potash is used instead of soda, so as to form a softer soap, and thus allow a more perfect saponification.

The saponification may also be conducted as follows: The alkali and fat in the melted state are shaken vigorously in the saponification flask until a complete emulsion is secured. The rest of the operation is then conducted as above.

(*c*) REMOVAL OF THE ALCOHOL.—(When method (b_1) has been used for saponification.)

The stoppers having been laid loosely in the mouth of the flask, the alcohol is removed by dipping the flask into a steam bath. The steam should cover the whole of the flask except the neck. After the alcohol is nearly removed, frothing may be noticed in the soap, and to avoid any loss from this cause or any creeping of the soap up the sides of the flask, it should be removed from the bath and shaken to and fro until the frothing disappears. The last traces of alcohol vapor may be removed from the flask by waving it briskly, mouth down, to and fro.

(*d*) DISSOLVING THE SOAP.—After the removal of the alcohol the soap should be dissolved by adding 100 cc of recently boiled distilled water, or 80 cc when method (b_2) has been used for saponification, warming on the steam bath with occasional shaking until solution of the soap is complete.

(*e*) SETTING FREE THE FATTY ACIDS.—When the soap solution has cooled to about 60° or 70°, the fatty acids are separated by adding 40 cc of the dilute sulfuric acid solution mentioned above, or 60 cc when method (b_2) has been used for saponification.

(*f*) MELTING THE FATTY ACID EMULSION.—The flask should now be restoppered as in the first instance, and the fatty acid emulsion melted by replacing the flask on the steam bath. According to the nature of the fat examined, the time required for the fusion of the fatty acid emulsions may vary from a few minutes to several hours.

(*g*) THE DISTILLATION.—After the fatty acids are completely melted, which can be determined by their forming a transparent oily layer on the surface of the water, the flask is cooled to room temperature, and a few pieces of pumice stone added. The pumice stone is prepared by throwing it, at a white heat, into distilled water, and keeping it under water until used The flask is now connected with a glass condenser, slowly heated with a naked flame until ebullition begins, and then the distillation continued by regulating the flame in such a way as to collect 110 cc of

the distillate in, as nearly as possible, thirty minutes. The distillate should be received in a flask accurately graded at 110 cc.

(*h*) TITRATION OF THE VOLATILE ACIDS.—The 110 cc of distillate, after thorough mixing, are filtered through perfectly dry filter paper, 100 cc of the filtered distillate poured into a beaker holding from 200 to 250 cc, 0.5 cc phenolphthalein solution added, and decinormal barium hydrate run in until a red color is produced. The contents of the beaker are then returned to the measuring flask to remove any acid remaining therein, poured again into the beaker, and the titration continued until

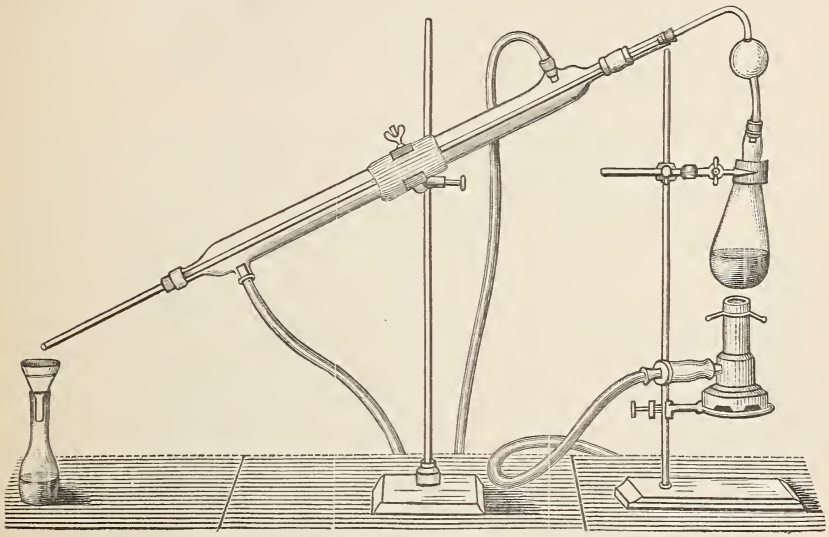

FIG. 2.—Apparatus for the distillation of volatile acids.

the red color produced remains apparently unchanged for two or three minutes. The number of cubic centimeters of decinormal barium hydroxid required should be increased by one-tenth.

(g) DETERMINATION OF SOLUBLE AND INSOLUBLE ACIDS.

(1) *Preparation of reagents.*

(*a*) STANDARD CAUSTIC SODA SOLUTION.—A decinormal solution of caustic soda is used. Each cubic centimeter contains 0.0040 gram of sodium hydroxid and neutralizes 0.0088 gram of butyric acid ($C_4H_8O_2$).

(*b*) ALCOHOLIC POTASH SOLUTION.—Dissolve 40 grams of good caustic potash in 1 liter of 95 per cent redistilled alcohol. The solution must be clear and the potassium hydroxid free from carbonates.

(*c*) STANDARD ACID SOLUTION.—Prepare accurately a half normal solution of hydrochloric acid.

(*d*) INDICATOR.—Dissolve 1 gram of phenolphthalein in 100 cc of 95 per cent alcohol.

(2) *Determination.*

(*a*) SOLUBLE ACID.—About 5 grams of the sample are weighed into a saponification flask (p. 27), 50 cc of the alcoholic potash solution added, and the flask stoppered and placed in the steam bath until the fat is entirely saponified. The operation may be facilitated by occasional agitation. The alcoholic potash is always measured with the same pipette, and uniformity further secured by allowing it to drain the same length of time (thirty seconds). Two or three blank experiments are conducted at the same time.

In from five to thirty minutes, according to the nature of the fat, the liquid will appear perfectly homogeneous, and when this is the case the saponification is complete and the flask is removed and cooled. When sufficiently cool, the stopper is removed and the contents of the flask rinsed with a little 95 per cent alcohol into an Erlenmeyer flask of about 200 cc capacity, which is placed on the steam bath together with the blanks until the alcohol is evaporated.

Titrate the blanks with half normal hydrochloric acid, using phenolphthalein as indicator. Then run into each of the flasks containing the fatty acids 1 cc more of the half normal hydrochloric acid than is required to neutralize the potash in the blanks. The flask is then connected with a condensing tube 3 feet long, made of small glass tubing, and placed on the steam bath until the separated fatty acids form a clear stratum on the upper surface of the liquid. The flask and contents are then cooled in ice water.

The fatty acids having quite solidified, the liquid contents of the flask are poured through a dry filter into a liter flask, care being taken not to break the cake. Between 200 and 300 cc of water are next brought into the flask, the cork with its condenser tube reinserted, and the flask placed on the steam bath until the cake of acids is thoroughly melted. During the melting of the cake of fatty acids the flask should occasionally be agitated with a circulatory motion in such a way that its contents are not made to touch the cork. When the fatty acids have again separated into an oily layer, the flask and its contents are cooled in ice water and the liquid filtered through the same filter into the same liter flask. This treatment with hot water, followed by cooling and filtration of the wash water, is repeated three times, the washings being added to the first filtrate. The mixed washings and filtrate are next made up to 1 liter, and 100 cc in duplicate are titrated with decinormal sodium hydroxid. The number of cubic centimeters of sodium hydroxid required for each 100 cc of the filtrate is multiplied by 10 to correct for the total volume. The number so obtained represents the volume of decinormal sodium hydroxid neutralized by the soluble fatty acids of the butter fat taken, plus that corresponding to the excess of the standard acid used, viz, 1 cc. The number is therefore to be diminished by 5, corresponding to the excess of 1 cc of half normal acid. This corrected volume multiplied by 0.0088 gives the weight of butyric acid in the amount of butter fat saponified.

(*b*) Insoluble acids.—The flask containing the cake of insoluble fatty acids from (*a*), and the paper through which the soluble fatty acids have been filtered, are allowed to drain and dry for twelve hours, when the cake, together with as much of the fatty acids as can be removed from the filter paper, are transferred to a weighed glass evaporating dish. The funnel, with the filter, is then placed in an Erlenmeyer flask, and the paper thoroughly washed with absolute alcohol. The flask is rinsed with the washings from the filter paper, then with pure alcohol, and these transferred to the evaporating dish. The dish is placed on the steam bath until the alcohol is evaporated, dried for two hours at 100°, cooled in a desiccator, and weighed. It is again placed in the air bath for another two hours, cooled as before, and weighed. If there be any considerable decrease in weight, reheat two hours and weigh again. This gives the weight of insoluble fatty acids in the quantity of substance taken, from which the percentage is easily calculated.

(h) Determination of saponification equivalent (Koettstorfer number).

(1) *Preparation of reagents.*

The reagents used in this determination are the same as those given under (g) (1), page 29, except that the standard caustic soda solution is not required.

(2) *Determination.*

Between 1 and 2 grams of the sample are weighed into a saponification flask (2) (*a*), page 27, 25 cc of the alcoholic potash solution added, and the flask stoppered and placed in the steam bath until the fat is entirely saponified. The operation

may be facilitated by occasional agitation. The alcoholic potash is always measured with the same pipette, and uniformity further secured by allowing it to drain the same length of time (thirty seconds). Two or three blank experiments are conducted at the same time. As soon as the saponification is complete, the flasks are removed from the bath, cooled, and the contents are titrated with the half normal hydrochloric acid, using phenolphthalein as indicator. The Koettstorfer number (milligrams of potassium hydroxid required to saponify 1 gram of the fat) is obtained by subtracting the number of cubic centimeters of hydrochloric acid necessary to neutralize the alkali after saponification from the number necessary to neutralize the blank, multiplying the result by 28.06, and dividing the product by the number of grams of fat used for the determination.

(i) DETERMINATION OF THE REFRACTIVE INDEX.

The refractive index is conveniently determined by Abbe's refractometer. A piece of fine tissue paper, 3 cm in length by 1.5 cm in width, is placed on the lower of the two glass prisms of the apparatus. Two or three drops of oil or fat are placed upon the paper, and the upper prism carefully fixed in position, so as not to move the paper from its place. In charging the apparatus with the oil in this way it is placed in a horizontal position. After the paper disk holding the fat is secured by replacing the upper prism the apparatus is placed in its normal position and the index moved until the light directed through the apparatus by the mirror shows the field of vision divided into dark and light portions. The dispersion apparatus is now turned until the rainbow colors on the part between the dark and light field have disappeared. Before doing this, however, the telescope, the eyepiece of the apparatus, is so adjusted as to bring the cross lines of the field of vision distinctly into focus. The index of the apparatus is now moved back and forth until the dark edge of the field of vision falls exactly in the intersection of the cross lines. The refractive index of the fat under examination is then read directly upon the scale by means of a small magnifying glass. To check the accuracy of the first reading, the dispersion apparatus should be turned through an angle of 180° until the colors have again disappeared, and the scale of the instrument again read. These two readings should nearly coincide, and their mean is the true reading of the fat under examination.

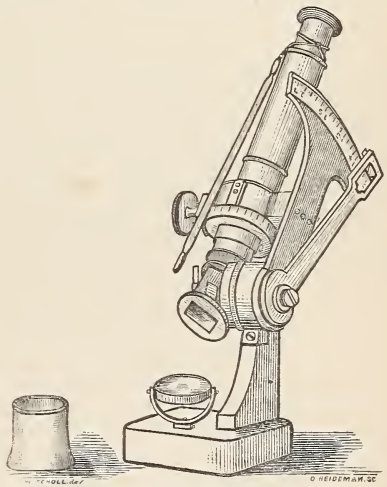

FIG. 3.—Abbe's refractometer.

For butter fats the apparatus should be kept in a warm place, the temperature of which does not fall below 30°. For reducing the results obtained to a standard temperature, say 25°, the factor 0.000176 may be used. As the temperature rises the refractive index falls.

Example.—Refractive index of a butter fat determined at 32.4° = 1.4540, reduced to 25° as follows: 32.4 — 25 = 7.4; 0.000176 × 7.4 = 0.0013; then 1.4540 + 0.0013 = 1.4553.

The instrument used should be set with distilled water at 25°, the theoretical refractive index of water at that temperature being 1.3330. In the determination above given, the refractive index of pure water measured 1.3300; hence the above numbers should be corrected for theory by the addition of 0.0030, making the corrected index of the butter fat mentioned at the temperature given 1.4583.

(j) DETERMINATION OF IODIN ABSORPTION NUMBER.

(1) *Preparation of reagents.*

(*a*) IODIN SOLUTION.—Dissolve 25 grams of pure iodin in 500 cc of 95 per cent alcohol. Dissolve 30 grams of mercuric chlorid in 500 cc of 95 per cent alcohol. The latter solution, if necessary, is filtered, and then the two solutions mixed. The mixed solution should be allowed to stand twelve hours before using.

(*b*) DECINORMAL SODIUM THIOSULFATE SOLUTION.—Dissolve 24.6 grams of chemically pure sodium thiosulfate freshly pulverized as finely as possible and dried between filter or blotting paper, and dilute with water to 1 liter at the temperature at which the titrations are to be made.

(*c*) STARCH PASTE.—One gram of starch is boiled in 200 cc of distilled water for ten minutes and cooled to room temperature.

(*d*) SOLUTION OF POTASSIUM IODID.—One hundred and fifty grams of potassium iodid are dissolved in water and made up to 1 liter.

(*e*) SOLUTION OF POTASSIUM DICHROMATE.—Dissolve 3.874 grams of chemically pure potassium dichromate in distilled water and make the volume up to 1 liter at the temperature at which the titrations are to be made.

(2) *Determination.*

(*a*) STANDARDIZING THE SODIUM THIOSULFATE SOLUTION.—Place 20 cc of the potassium-dichromate solution, to which has been added 10 cc of the solution of potassium iodid, in a glass-stoppered flask. Add to this 5 cc of strong hydrochloric acid. Allow the solution of sodium thiosulfate to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste, and with constant shaking continue to add the sodium thiosulfate solution until the blue color just disappears. The number of cubic centimeters of thiosulfate solution used multiplied by 5 is equivalent to 1 gram of iodin.

Example.—Twenty cubic centimeters $K_2Cr_2O_7$ solution required 16.2 cc sodium thiosulfate; then $16.2 \times 5 = 81$ = number cubic centimeters of thiosulfate solution equivalent to 1 gram of iodin. Then 1 cc thiosulfate solution = 0.0124 gram of iodin. Theory for decinormal solution of sodium thiosulfate, 1 cc = 0.0127 gram of iodin.

(*b*) WEIGHING THE SAMPLE.—About 1 gram of butter fat is weighed in a glass-stoppered flask holding about 300 cc, with the precautions mentioned for weighing the fat for determining volatile acids (f) (3), page 27.

(*c*) ABSORPTION OF IODIN.—The fat in the flask is dissolved in 10 cc of chloroform. After complete solution has taken place, 30 cc of the iodin solution (1) (*a*) are added. The flask is now placed in a dark place and allowed to stand, with occasional shaking, for three hours.

(*d*) TITRATION OF THE UNABSORBED IODIN.—One hundred cc of distilled water are added to the contents of the flask, together with 20 cc of the potassium iodid solution. Any iodin which may be noticed upon the stopper of the flask should be washed back into the flask with the potassium iodid solution. The excess of iodin is taken up with the sodium thiosulfate solution, which is run in gradually, with constant shaking, until the yellow color of the solution has almost disappeared. A few drops of starch paste are added, and the titration continued until the blue color has entirely disappeared. Toward the end of the reaction the flask should be stoppered and violently shaken, so that any iodin remaining in solution in the chloroform may be taken up by the potassium iodid solution in the water. A sufficient quantity of sodium thiosulfate solution should be added to prevent a reappearance of any blue color in the flask for five minutes.

(*e*) SETTING THE VALUE OF THE IODIN SOLUTION BY THE THIOSULFATE SOLUTION.—At the time of adding the iodin solution to the fat, two flasks of the same size as those used for the determination should be employed for conducting the operation

described above, but without the presence of any fat. In every other respect the performance of the blank experiments should be just as described. These blank experiments must be made each time the iodin solution is used.

Example blank determinations.—Thirty cc iodin solution required 46.4 cc of sodium thiosulfate solution. Thirty cc iodin solution required 46.8 cc of sodium thiosulfate solution. Mean, 46.6 cc.

Per cent of iodin absorbed:

Weight of fat taken	grams	1.0479
Quantity of iodin solution used	cubic centimeters	30.0
Thiosulfate equivalent to iodin used	do	46.6
Thiosulfate equivalent to remaining iodin	do	14.7
Thiosulfate equivalent to iodin absorbed	do	31.9

Per cent of iodin absorbed, $31.9 \times 0.0124 \times 100 \div 1.0479 = 37.75$.

(k) DETERMINATION OF SPECIFIC GRAVITY.

(1) *Standardization of flasks.*

Use a small specific gravity flask of from 25 to 30 cc capacity. The flask is to be thoroughly washed with hot water, alcohol, and ether, and then dried. After cooling in a desiccator, the weight of the flask and stopper is accurately determined.

The flask is filled with freshly boiled and still hot distilled water, and placed in a bath of pure distilled water. The water of the bath is kept in brisk ebullition for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. The stopper, previously heated to 100°, is then inserted, the flask removed, wiped dry, and after it has nearly cooled to room temperature placed in the balance, and weighed when balance temperature is reached.

OPTIONAL METHOD OF STANDARDIZING FLASKS.—The following formula may be used for calculating the volume V, in cubic centimeters, of a glass vessel from the weight P of water at temperature t contained therein, and the volume V' at any other temperature t':

$$V = P\frac{p}{d}$$

$$V' = P\frac{p}{d}\left[1 + \gamma\,(t' - t)\right]$$

p = weight (in brass weights) of 1 cc of water in vacuo. This is so nearly 1 that it will not affect the result in the fifth place of decimals, and may therefore be disregarded. Hence the formula stands:

$$V' = P\frac{1}{d}\left[1 + \gamma\,(t' - t)\right]$$

d = density of water at temperature t.

$\gamma = 0.000025$, the cubical expansion coefficient of glass.

(2) *Determination.*

WEIGHT OF FAT AT THE TEMPERATURE OF BOILING WATER.—The flask is rinsed with alcohol and ether, and dried for a few minutes at the temperature of boiling water. It is filled with the dry, hot, fresh-filtered fat, which should be entirely free from air bubbles, replaced in the water bath, and kept for thirty minutes at the temperature of boiling water. The stopper, previously heated to 100°, is inserted, the flask removed, wiped dry, placed in the balance after it has nearly cooled to room temperature, and weighed when the balance temperature is reached. The weight of fat having been determined, the specific gravity is obtained by dividing it by the weight of water previously found.

Example:

	Grams.
Weight of flask, dry	10.0197
Weight of flask, plus water	37.3412
Weight of water	27.3215
Weight of flask, plus fat	34.6111
Weight of fat	24.5914

Specific gravity = 24.5914 ÷ 27.3215 = 0.90008.

The weight of the flask dry and empty, and the weight of water at boiling temperature contained therein, may be used constantly if great care be taken in handling and cleaning the apparatus.

Example:

	Grams.
Weight of flask, dry and empty	10.0028
Weight of flask after three weeks' use	10.0030

(1) DETERMINATION OF MELTING POINT.

(1) *Preparation of reagents.*

(*a*) A piece of ice floating in distilled water that has been recently boiled.

(*b*) A mixture of alcohol and water of the same specific gravity as the fat to be examined. This is prepared by boiling distilled water and 95 per cent alcohol for ten minutes to remove the gases which they may hold in solution. While still hot, the water is poured into the test tube described below ((2)(*e*)) until it is nearly half full. The test tube is nearly filled with the hot alcohol, which is carefully poured down the side of the inclined tube to avoid too much mixing. If the alcohol is not added until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top.

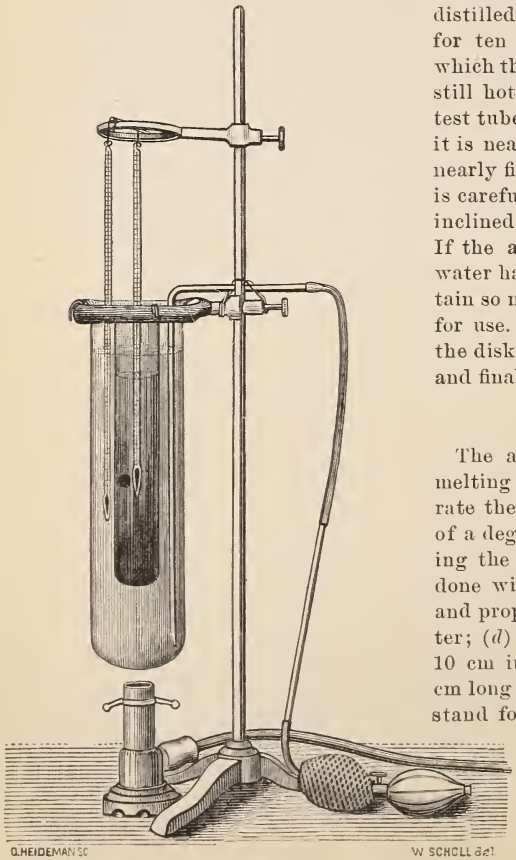

FIG. 4.—Apparatus for the determination of melting point.

(2) *Apparatus.*

The apparatus for determining the melting point consists of (*a*) an accurate thermometer reading easily tenths of a degree; (*b*) a cathetometer for reading the thermometer (but this may be done with an eyeglass if held steadily, and properly adjusted); (*c*) a thermometer; (*d*) a tall beaker 35 cm high and 10 cm in diameter; (*e*) a test tube 30 cm long and 3.5 cm in diameter; (*f*) a stand for supporting the apparatus; (*g*) some method of stirring the water in the beaker (for example, a blowing bulb of rubber, and a bent glass tube extending to near the bottom of the beaker).

(3) *Determination.*

The disks of fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of from 15 to 20 cm on a smooth piece of ice floating in distilled water that has been recently boiled. The disks thus formed are from 1 to 1.5 cm in diameter, and weigh about 200 mg. By pressing the ice under the water the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The test tube containing the alcohol and water is placed in a tall beaker containing water and ice, until cold. The disk of fat is then dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk, the thermometer is moved from time to time in a circularly pendulous manner.

The disk having been placed in position, the water in the beaker is slowly heated, and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° below the melting point, the disk of fat begins to shrivel, and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotatory movement should be given to the thermometer bulb. The rise of temperature should be so regulated that the last 2° of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so the reading of the thermometer is to be made. As soon as the temperature is taken the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test tube (ice water having been used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum of about 1.5° above the melting point of the fat under examination.

The edge of the disk should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place, and the disk adhere to the sides of the tube, a new trial should be made.

Triplicate determinations should be made, and the second and third results should show a near agreement.

Example.—Melting point of sample of butter:

	Degrees.
First trial	33.15
Second trial	33.05
Third trial	33.00

(m) MICROSCOPIC EXAMINATION.

Place a small portion of the fresh samples, taken from the inside of the mass, on a slide, add a drop of pure sweet oil, cover with gentle pressure, and examine with a one-half to one-eighth inch objective for crystals of lard, etc. Examine the same specimen with polarized light and a selenite plate without the use of oil. Pure fresh butter will neither show crystals nor a particolored field with selenite. Other fats melted and cooled and mixed with butter will usually present crystals and variegated colors with the selenite plate.

For further microscopic study dissolve from 4 to 5 cc of the fat in 15 cc of ether in a test tube. Close the tube with a loose plug of cotton wool and allow to stand

from twelve to twenty-four hours at room temperature (20° to 25°). When crystals form at the bottom of the tube, they are removed with a pipette, glass rod, or tube, placed on a slide, covered, and examined. The crystals formed by later deposits may be examined in a similar way.

2. Milk Analysis.

(a) determination of water.

Evaporate from 1 to 2 grams of milk in a tared flat dish containing from 15 to 20 grams of pure dry sand, or without sand, until apparently dry. Dry and heat for one hour at the temperature of boiling water. Cool in a desiccator, and weigh rapidly to avoid absorption of hygroscopic moisture.

Babcock asbestus method.—Provide a hollow cylinder of perforated sheet metal, 60 mm long and 20 mm in diameter, closed 5 mm from one end by a disk of the same material. The perforations should be about 0.7 mm in diameter and about 0.7 mm apart. Fill loosely with from 1.5 to 2.5 grams of freshly ignited, woolly asbestus, free from fine and brittle material, cool in a desiccator, and weigh. Introduce a weighed quantity of milk (between 3 and 5 grams) and dry at 100° to constant weight for the determination of total solids.

(b) determination of ether extract.

If the milk be sour, about 10 per cent of strong ammonia water and a small quantity of anhydrous sodium carbonate should be added before evaporation, and the results corrected for the volume of the ammonia added.

(1) *Babcock asbestus method.*

Extract the residue from the determination of water by the Babcock asbestus method with anhydrous ether until the fat is removed, evaporate the ether, dry the fat at 100°, and weigh. The fat may also be determined by difference, drying the extracted cylinders at 100°.

(2) *Paper-coil method.*

Coils made of thick filter paper, cut into strips 6.25 by 62.5 cm, are thoroughly extracted with ether and alcohol, or the weight of the extract corrected by a constant obtained for the paper. If this latter method is used, a small amount of anhydrous sodium carbonate should be added. From a weighing bottle about 5 grams of milk are transferred to the coil by a pipette, care being taken to keep the end of the coil held in the fingers dry. The coil, dry end down, on a piece of glass, is dried at the temperature of boiling water for one hour, or, better, dried in hydrogen at the temperature of boiling water, transferred to an extraction apparatus, and extracted with absolute ether or petroleum ether boiling at about 45°. The extracted fat is dried in hydrogen and weighed.

(c) determination of total nitrogen.

Place in Kjeldahl digestion flask a known weight (about 5 grams) of milk, and proceed, without evaporation, exactly as described for this method under nitrogen, page 15. Multiply the percentage of nitrogen by 6.25 for nitrogen compounds.

(d) determination of ash.

In a weighed dish put 20 cc of milk from a weighing bottle; add 6 cc of nitric acid, evaporate to dryness, and burn at a low red heat until the ash is free from carbon.

(e) determination of sugar.

See under Determination of Carbohydrates in Agricultural Products, pages VI, 6.

3. Cheese Analysis (Provisional Methods).

(a) preparation of sample.

Where the cheese can be cut, a narrow wedge reaching from the edge to the center of the cheese will more nearly represent the average composition than any other sample. This should be chopped quite fine, with care to avoid evaporation of water, and the several portions for analysis taken from the mixed mass. When the sample is taken with a cheese trier, a plug taken perpendicular to the surface one-third of the distance from the edge to the center of the cheese more nearly represents the average composition than any other. The plug should either reach entirely through or only half way through the cheese. For inspection purposes the rind may be rejected, but for investigations where the absolute quantity of fat in the cheese is required the rind should be included in the sample. It is well, when admissible, to take two or three plugs on different sides of the cheese, and, after splitting them lengthwise with a sharp knife, take portions of each for the different determinations.

(b) determination of water.

From 5 to 10 grams of cheese should be taken, and placed in thin slices in a weighed platinum or porcelain dish which contains a small quantity of freshly ignited asbestus, to absorb the fat which may run out of the cheese. This is then heated in a water oven for ten hours and weighed; the loss in weight is considered as water. Or, if preferred, the dish may be placed in a desiccator over concentrated sulfuric acid and dried to constant weight. In some cases this may require as much as two months. The acid should be renewed when the cheese has become nearly dry.

(c) determination of ether extract.

Grind from 5 to 10 grams of cheese in a small mortar with about twice its weight of anhydrous copper sulfate. The grinding should continue until the cheese is finely pulverized and evenly distributed throughout the mass, which will have a uniform light blue color. This mixture is transferred to a glass tube which has a strong filter paper, supported by a piece of muslin, tied over one end. Put a little anhydrous copper sulfate into the tube next to the filter before introducing the mixture containing the cheese. On top of the mixture place a tuft of ignited asbestus, and place the tube in a continuous extraction apparatus and extract with anhydrous ether for fifteen hours. Dry the fat obtained at 100° to constant weight.

(d) determination of nitrogen.

Make a determination of nitrogen by the Kjeldahl method, using about 2 grams of cheese, and multiply the percentage of nitrogen found by 6.25 for nitrogen compounds.

(e) determination of ash.

The dry residue from the water determination may be taken for the ash. If the cheese be rich in fat, the asbestus will be saturated therewith. This may be ignited carefully and the fat allowed to burn off, the asbestus acting as a wick. No extra heat should be applied during this operation, as there is danger of spurting. When the flame has died out, the burning may be completed in a muffle at low redness. When desired, the salt may be determined in the ash in the manner specified under butter, analysis, page 26.

(f) determination of other constituents.

The sum of the percentages of the different constituents, determined as above, subtracted from 100 will give the amount of organic acids, milk sugar, etc., in the cheese.

IV.—METHODS FOR THE ANALYSIS OF SOILS.

1. Preparation of Sample.

Surface accumulations of decaying leaves, etc., should be removed and a slice of uniform thickness from the surface to the desired depth should be taken. To eliminate accidental variations in the soil, select specimens from five or six places in the field and remove several pounds of the soil, taking it to the depth of 6 inches, or to the change between the surface soil and the subsoil, in case such change occurs between the depth of 6 and 12 inches. In no case is the sample to be taken to a greater depth than 12 inches. If the surface soil extend to a greater depth, a separate sample below the depth of 12 inches is to be taken. If the surface soil extend to a depth of less than 6 inches, and the difference between it and the subsoil is unusually great, a separate sample of the surface soil should be taken besides the one to the depth of 6 inches.

The depth to which the sample of subsoil should be taken will depend on circumstances. It is always necessary to know what constitutes the foundation of a soil, to the depth of 3 feet at least, since the question of drainage, resistance to drought, etc., will depend essentially upon the nature of the substratum. But in ordinary cases 10 or 12 inches of subsoil will be sufficient for the purposes of examination in the laboratory. The specimen should be taken in other respects precisely like that of the surface soil, while that of the material underlying this subsoil may be taken with less exactness, perhaps at some ditch or other easily accessible point, and should not be broken up, but left, as nearly as possible, in its original state. Mix these soils intimately, remove any stones, shake out all roots and foreign matters, expose in thin layers in a warm room till thoroughly air dry, or dry it in an air bath at a temperature of 40°.

The soil is rapidly dried to arrest nitrification. It is not heated above 40° lest there be dissipation of ammonium compounds, or a change in the solubility of the soil. The normal limit to which the soil may be heated in place by the sun's rays should not be exceeded in preparing a soil for an agricultural chemical analysis.

Five hundred grams or more of the air-dried soil, which may be either the original soil or that which has been passed through a sieve of coarser mesh, are sifted through a sieve with circular openings one-half millimeter in diameter, rubbing, if necessary, with a rubber pestle in a mortar until the fine earth has been separated as completely as possible from the particles that are too coarse to pass the sieve. The fine earth is thoroughly mixed and preserved in a tightly stoppered bottle, from which the portions for analysis are weighed.

The coarse part is weighed and examined microscopically or with Thoulet's solution.

It may sometimes be necessary to wash the soil through the one-half millimeter sieve with water; but this is to be avoided whenever possible.

2. Determination of Moisture.

Heat from 2 to 5 grams of the air-dried soil in a flat-bottomed, tared platinum dish for five hours in a water oven kept briskly boiling; cover the dish, cool in a desiccator, and weigh. Repeat the heating, cooling, and weighing at intervals of two hours till nearly constant weight is found, and estimate the moisture by the loss of weight. Weigh rapidly, to avoid absorption of moisture from the air. An air bath must not be used in this determination.

3. Determination of Volatile Matter.

The platinum dish and soil used to determine moisture are used also to determine volatile matter. Heat the dish and dry soil to full redness, until all organic matter is burned away. If the soil contain appreciable quantities of carbonates, the con-

tents of the dish, after cooling, are moistened with a few drops of a saturated solution of ammonium carbonate, dried and heated to dull redness to expel salts of ammonium, cooled in the desiccator, and weighed. The loss in weight represents the organic matter, water of combination, salts of ammonium, etc.

4. Determination of Acid-Soluble Materials.

In the following scheme for soil analysis it is intended to use the air-dried soil from the sample bottle for each separate investigation. The determination of moisture, made once for all on a separate portion of air-dried soil, will afford the datum for calculating the results of analysis upon the soil dried at the temperature of boiling water. It is not desirable to ignite the soil before analysis, or to heat it so as to change its chemical properties.

The acid digestion is to be performed in a flask so arranged that the evaporation of acid shall be reduced to a minimum, but under atmospheric pressure and at the temperature of boiling water. The digestion is easily accomplished in a flat-bottomed conical flask of hard glass, carrying a stopper and hard-glass condensing tube at least 18 inches long. Where sulfuric acid is to be determined, a rubber stopper can not be used. A flask with ground glass stopper, carrying a condensing tube, is useful in such cases.

The flask must be immersed in the water bath up to the neck, or at least to the level of the acid, and the water must be kept boiling continuously during the digestion.

In the following scheme 10 grams of soil are used, this being a convenient quantity of most soils, in which the insoluble matter is about 80 per cent. If desired, a larger quantity of such soil may be used, with a proportionately larger quantity of acid, and making up the soil solution to a proportionately larger volume. In very sandy soils, where the proportion of insoluble matter is 90 per cent or more, 20 grams of soil are to be digested with 100 cc of acid and the solution made up to 500 cc; or a larger quantity may be used, preserving the same proportions. It is very important that the analyst assure himself of the purity of all the reagents to be used in the analysis of soils before beginning the work.

(a) Acid digestion of the soil.

Place 10 grams of the air-dried soil in an Erlenmeyer glass flask of from 150 to 200 cc capacity, add 100 cc of pure hydrochloric acid of specific gravity 1.115, insert the stopper with condensing tube, place in a water or steam bath, and digest for ten hours continuously at the temperature of boiling water, shaking once each hour. Pour the clear liquid from the flask into a small beaker and wash the residue out of the flask with distilled water on a filter, adding the washings to the contents of the beaker. The residue, after washing until free of acid, is dried and ignited, as directed below. Oxidize the organic matter present in the filtrate with nitric acid and evaporate to dryness on the water bath, finishing on a sand or air bath to complete dryness; take up with hot water and a few cubic centimeters of hydrochloric acid and again evaporate to complete dryness. Take up as before, filter, and wash thoroughly with cold water, or with hot water slightly acidified at first with hydrochloric acid. Cool and make up to 500 cc. This is Solution A. The residue is to be added to the main residue and the whole ignited and weighed, giving the "insoluble matter." (See 5, p. 42.)

(b) Determination of ferric oxid, alumina, and phosphoric acid, collectively.

To 100 or 200 cc of Solution A, according to the probable amount of iron and alumina present, add ammonium hydroxid to slightly alkaline reaction to precipitate ferric and aluminic hydrates and phosphates. Expel the excess of ammonia by boiling, allow to settle, and decant the clear solution through a filter; add to the flask

50 cc of hot distilled water, boil, settle, and decant as before. After pouring off all the clear solution possible, dissolve the residue with a few drops of hydrochloric acid and precipitate again with ammonium hydroxid exactly as before; transfer all the precipitate to the filter and wash with hot distilled water till the washings become free from chlorids. Save the filtrates and washings which form Solution B. Dry the filter and precipitate, transfer the precipitate to a tared platinum crucible, burn the filter, and add the ash to the precipitate; ignite to bright redness, cool in a desiccator, and weigh. The increase of weight, minus the ash of filter and the phosphoric acid (found in a separate process), represents the weight of the Fe_2O_3 and Al_2O_3.

(c) DETERMINATION OF MANGANESE.

Concentrate the filtrates and washings (Solution B) to 100 cc or less; add ammonium hydroxid to alkalinity; add bromin water and heat to boiling, keeping the beaker covered with a watch crystal; as the bromin escapes the beaker is allowed to cool somewhat, more ammonia and bromin water being added and heated as before. This process is continued until the manganese is completely precipitated, which requires from fifteen to thirty minutes. The solution is then to be slightly acidified with a few drops of acetic acid and filtered while still boiling hot, the precipitate washed with hot water, dried, ignited, and weighed as Mn_3O_4.

(d) DETERMINATION OF CALCIUM.

If no manganese be precipitated, evaporate Solution B or the filtrates and washings from (c) to about 50 cc, make slightly alkaline with ammonia, and add, while still hot, ammonium oxalate solution as long as any precipitate is produced, adding a few cubic centimeters in excess to convert the magnesium also into oxalate. Heat to boiling, allow the precipitate to settle, decant the clear solution on a filter, pour from 15 to 20 cc of hot distilled water on the precipitate, and again decant the clear solution on the filter. Dissolve the precipitate in the beaker with a few drops of hydrochloric acid, add a little water, and reprecipitate, boiling hot, by adding ammonium hydroxid to slight alkalinity and a little ammonium oxalate solution; filter through the same filter, transfer the precipitate to the filter, and wash it free from chlorids. Dry, ignite the precipitate over the blast lamp until it ceases to lose weight, weigh, and estimate as CaO.

(e) DETERMINATION OF MAGNESIUM.

Acidify the filtrate and washings from (d) slightly with hydrochloric acid and concentrate to about 50 cc, place in a small Erlenmeyer flask or beaker, make slightly alkaline with ammonium hydroxid, and add sufficient acid sodium phosphate solution to precipitate the magnesium; then add gradually 10 cc strong ammonium hydroxid, cover closely to prevent escape of ammonium, and let stand in the cold. Filter after twelve hours, wash the precipitate free from chlorids, dry, burn at first at a moderate heat, finally igniting intensely, and weigh as $Mg_2P_2O_7$.

(f) DETERMINATION OF FERRIC OXID.

Evaporate 100 cc of Solution A, with the addition of about 10 cc of sulfuric acid, until all hydrochloric acid is expelled; dilute with water, reduce with zinc, and estimate ferric oxid by a standard solution of potassium permanganate. To prepare potassium permanganate solution, dissolve 3.156 grams of pure crystallized potassium permanganate in 2,000 cc of distilled water, and preserve in a glass-stoppered bottle, shielded from the light. Standardize this solution after it has stood twenty-four hours with pure ammonio-ferrous sulfate, oxalic acid, or metallic iron.

Instead of using another portion of the solution, the weighed precipitate from (b) may be dissolved by digestion on the water bath in a covered beaker or flask with from 10 to 20 cc of a mixture of one part H_2SO_4 with four parts of water.

Deduct the per cent of ferric oxid obtained from the per cent of ferric oxid and alumina (b), and make corrections for filter ash and phosphoric acid, to obtain the per cent of alumina.

(g) DETERMINATION OF PHOSPHORIC ACID.

Evaporate 100 or 200 cc of Solution A to about 25 or 30 cc on the water bath; nearly neutralize with ammonium hydroxid, add about 10 grams pure crystallized ammonium nitrate, and gradually about 20 cc molybdic solution ((1) (*b*), p. 12) and set in water bath at a temperature of 40°. When the precipitate has settled sufficiently, draw out with a pipette about 5 cc of the clear liquid, and test it by allowing it to run into 5 cc of warm molybdic solution. If any precipitate be produced, the test liquid is returned to the main portion and more molybdic solution is added and the operation repeated until all the phosphoric acid is precipitated. After standing from eight to twelve hours at a temperature not above 40°, the ammonium phosphomolybdate is filtered off and the phosphoric acid determined as magnesium pyrophosphate, as directed under Total Phosphoric Acid in Fertilizers, page 12. It is recommended to redissolve the magnesium ammonium phosphate precipitate in acetic acid, after it has been washed once or twice, and reprecipitate with ammonia and a fresh quantity of magnesia mixture, giving the usual time for the separation of the precipitate. If there be any residue of phosphates remaining on dissolving the phosphomolybdate in ammonia, or the magnesium ammonium phosphate in acetic acid, this residue is dissolved in a little hydrochloric acid, neutralized with ammonium hydroxid and precipitated with molybdic solution, and the phosphomolybdate obtained added to the main quantity.

(h) DETERMINATION OF SULFURIC ACID.

Evaporate 100 or 200 cc of Solution A nearly to dryness on a water bath to expel the excess of acid; then add 50 cc of distilled water; heat to boiling and add from 2 to 3 cc of a solution of barium chlorid, and continue the boiling for five minutes. When the precipitate is settled, pour the liquid on a tared Gooch filter, treat the precipitate with from 15 to 20 cc of boiling water, and transfer the precipitate to the filter and wash with boiling water, at first slightly acidified with a few drops of hydrochloric acid, finally with pure water, till the filtrate is free from chlorids. Dry the filter and ignite strongly, and weigh as barium sulfate, which multiplied by 0.34331 equals SO_3.

(i) DETERMINATION OF POTASH AND SODA.

Treat the filtrate from (h) with ammonium hydroxid exactly as in (b). Evaporate the filtrate and washings to dryness, heat below redness, until ammonium salts are expelled, dissolve in about 25 cc of hot water, add 5 cc of baryta water and heat to boiling; let settle a few minutes and test a little of the clear liquid with more baryta water to be sure that enough has been added. When no further precipitate is produced, filter and wash thoroughly with hot water. Add ammonia and ammonium carbonate to complete the precipitation of the barium, let stand a short time on the water bath, filter and wash the precipitate thoroughly with hot water, evaporate filtrate and washings to dryness in a porcelain dish, expel ammonium salts by heat below redness, take up with a little hot water, add a few drops of ammonium hydroxid and a drop or two of ammonium carbonate, let stand a few minutes on the water bath, filter into a tared platinum dish, evaporate to dryness on the water bath and heat to dull redness, until all ammonium salts are expelled and the residue is nearly or quite white. The heat must not be sufficient to fuse the residue. The weight of the residue represents potassium and sodium chlorids. Determine the potassium present with platinum chlorid in the usual manner. The sodium chlorid is obtained by subtracting potassium chlorid thus found from the total weight obtained above.

Instead of this, a fresh aliquot portion of Solution A may be evaporated to dryness, redissolved in water and treated directly with milk of lime as in ash analysis, but without previous addition of barium chlorid.

5. Determination of Acid-Insoluble Materials.

The residue from 4 (a) may be analyzed by the usual methods for silicates. If it is desired to determine the silica soluble in alkalies, the residue must be dried at 100° and an aliquot portion removed before ignition, for treatment with sodium carbonate solution, as described under Ash Analysis, page 44. Another aliquot portion, or the rest of the residue, is ignited and weighed.

6. Determination of Total Alkalies.

Determine in a separate portion of the soil by J. Lawrence Smith's method, given in Crookes's Select Methods, second edition, pages 28–40; or, preferably, determine by this method the alkalies in the insoluble residue from 4 (a) and add the amount obtained from the hydrochloric acid solution.

7. Identification of Lithium, Cæsium, and Rubidium.

The salts of these elements are occasionally found in very small amounts in soils. Their agricultural uses are still in question, and their amount is too small to admit of quantitative estimation. A qualitative examination may be made by the spectroscope with the water-soluble materials evaporated to dryness and dissolved with two or three drops of hydrochloric acid or with the alkaline chlorids separated as in 4 (i) or 6.

8. Determination of Total Nitrogen.

Nitrogen is determined by the Kjeldahl method modified to include nitrates, as described in the methods for determining nitrogen (p. 17), using an appropriate weight of the sample. When the digestion is completed, water is added and the solution filtered or decanted from the insoluble residue and the residue washed with more water. The filtrate and washings are treated in the usual manner for determination of the ammonia. Unless the amount of nitrates present is considerable, it is not necessary to use the modified method. The amount of nitrate present in ordinary soils is so small that the simple Kjeldahl method (p. 15), carried out as just described, omitting the salicylic acid and thiosulfate, is perfectly satisfactory.

9. Determination of Carbon Dioxid.

Determine as in Ash Analysis, page 45, using from 5 to 10 grams of the sample.

10. Determination of Humus.

Ten grams of the sample are placed in a Gooch crucible, extracted with 1 per cent hydrochloric acid until the filtrate gives no reaction with ammonia and ammonium oxalate, and the acid removed by washing with water. The contents of the crucible (including the asbestus filter) are then washed into a glass-stoppered cylinder with 500 cc of 4 per cent ammonia and allowed to remain, with occasional shaking, for twenty-four hours. During this time the cylinder is inclined as much as possible without bringing the contents in contact with the stopper, thus allowing the soil to settle on the side of the cylinder, and exposing a very large surface to the action of the ammonia. The cylinder is then placed in a vertical position and left for twelve hours, to allow the sediment to settle to the bottom. The supernatant liquid is filtered and an aliquot portion evaporated, dried at 100°, and weighed. The residue is then ignited and again weighed. The humus is calculated from the difference in weights between the dried and the ignited residues.

11. Statement of Results.

All results of soil analysis are to be calculated as per cent of the soil dried to constant weight in the water oven (see Determination of Moisture, p. 38), and are to be stated in the following order:

Insoluble matter }
Soluble silica }
Potash (K_2O)
Soda (Na_2O)
Lime (CaO)
Magnesia (MgO)
Manganese oxid (MnO)
Ferric oxid (Fe_2O_3)
Alumina (Al_2O_3)
Phosphorus pentoxid (P_2O_5)
Sulfur trioxid (SO_3)
Carbon dioxid (CO_2)
Water and organic matter

Total
Humus
Ash
Phosphorus pentoxid
Silica
Nitrogen (organic)
Hygroscopic moisture
Moisture absorbed at —°

V.—METHODS FOR THE ANALYSIS OF ASHES.

1. Preparation of the Ash.

Before combustion the material must be thoroughly cleaned from all foreign matter, especially from adhering soil. The combustion should be carried on at a comparatively low temperature, never reaching a full red heat, because of the danger of volatilizing alkaline chlorids, etc., and of fusing the ash; nor in a strong draft of air, lest the lighter part of the ash be carried away. Combustion is best carried on in a flat platinum dish in a muffle. With substances rich in silica and alkalies it is better to first char the substance, wash with distilled water to remove soluble salts, then dry and incinerate the residue. Evaporate the aqueous extract and add this to the rest of the ash. With substances rich in phosphates, e. g., seeds and animal substances, char the material, remove salts by acetic acid, decant the acetic solution, wash with distilled water, and then complete the combustion. Add the acetic solution and washings to the final ash, evaporate to dryness, and gently ignite the whole to decompose the acetates. In whatever way obtained, the whole of the ash should be pulverized and intimately mixed while still warm, and preserved in a tight, dry bottle for analysis. If after incineration the ash has absorbed moisture, dry thoroughly at low redness before bottling. It is intended that the preliminary preparation of the ash shall bring it to a perfectly dry condition, rendering a moisture determination unnecessary, and that the portions for analysis shall be weighed from the prepared sample in this condition. As it is sometimes difficult or impossible to prepare an ash for analysis in large quantity, the following method has been arranged so that a small amount of ash may be used. Where the ash is to be had in abundance, larger quantities and more portions may be used, but the proportion of acid to substance taken is to be preserved. Much latitude must be left to individual judgment in adapting the method to particular cases.

2. Analysis of the Ash.

Five grams of the ash are treated in a beaker, covered with a watch glass, with 50 cc of hydrochloric acid of specific gravity 1.115, and digested on the water bath until all effervescence has ceased. The cover is then removed and the liquid evaporated to complete dryness to render the silica insoluble. The residue is moistened with from 5 to 10 cc of hydrochloric acid, taken up with about 50 cc of water, allowed to stand on the water bath a few minutes, and filtered through an S. & S. "hardened" filter, and thoroughly washed. The filtrate and washings are made up to 250 cc for analysis. This is Solution A.

(a) Determination of Carbon, Sand, and Silica.

The residue is washed from the filter (which may be used again) as completely as possible into a platinum dish, and boiled about five minutes with about 20 cc of a saturated solution of pure sodium carbonate; a few drops of pure sodium hydroxid solution are added and the liquid allowed to settle, and decanted through a tared Gooch filter. The residue is boiled with sodium carbonate solution and decanted as before a second and a third time, and is finally brought upon the filter and thoroughly washed, first with hot water, then with a little dilute hydrochloric acid, and finally with hot water until free from chlorids. The residue in the Gooch filter is dried at 110° to constant weight, giving the carbon and sand. It is then incinerated and the weight of the sand determined, the difference giving the carbon. It is advisable to examine the sand with a microscope to determine if it be really sand. The alkaline filtrate and washings from the carbon and sand are acidified with hydrochloric acid, evaporated to dryness, and the silica separated and determined in the usual way.

Instead of determining soluble silica directly from the sodium carbonate solution, as above, another portion of the ash may be treated with hydrochloric acid and evaporated to dryness as before described, filtered on an ordinary filter, washed, burned, and weighed, giving the weight of silica plus sand, from which the weight of sand is deducted to obtain soluble silica. It is inadmissible to separate the soluble silica from the residue after it has been ignited.

(b) Determination of Manganese, Calcium, and Magnesium.

Fifty or 100 cc of Solution A are neutralized with ammonia and a gram or two of sodium acetate and a few drops of acetic acid are added; boil for one or two minutes, filter, and wash the precipitate thoroughly with boiling water. Evaporate the filtrate and washings to about 50 cc, filter out any slight additional precipitate that may form, and determine manganese, calcium, and magnesium exactly as described in the analysis of soils. If the phosphoric acid be in excess of the iron and aluminum some ferric chlorid solution must be added before precipitating with sodium acetate.

(c) Determination of Phosphoric Acid.

Twenty-five or 50 cc of Solution A are neutralized with ammonia, ammonium nitrate added, and the phosphoric acid precipitated with molybdic solution exactly as described in the Methods for Total Phosphoric Acid in Fertilizers, page 12.

(d) Determination of Sulfuric Acid and Alkalies.

Twenty-five or 50 cc of Solution A are heated to boiling and barium chlorid solution added in small quantity until no further precipitate is produced. Let stand on the water bath until clear, and filter the barium sulfate on a tared Gooch filter, wash thoroughly with hot water, at first slightly acidified with hydrochloric acid, finally with pure water until free from chlorids, dry, ignite the barium sulfate, and weigh.

Evaporate the filtrate and washings in a porcelain dish to dryness, redissolve in about 50 cc of water, and add milk of lime, or barium hydroxid solution, which must

be perfectly free from alkalies, until no further precipitation is produced and it is evident that there is an excess of calcium or barium hydroxid present; boil for two or three minutes, filter hot, wash thoroughly with boiling water, precipitate the lime and baryta from the filtrate with ammonia and ammonium carbonate, filter, evaporate the filtrate to dryness in a porcelain dish, and drive off the ammonium salts by heat below redness. When cold, redissolve in from 15 to 20 cc of water, precipitate with a few drops of ammonia and ammonium carbonate solution, let stand a few minutes on the water bath, filter into a tared platinum dish, evaporate to dryness, expel the ammonium salts by heating to just perceptible dull redness, weigh the potassium and sodium chlorids obtained, and determine the potassium with platinum chlorid.

(e) DETERMINATION OF CARBON DIOXID.

Use from 1 to 5 grams of ash in any of the usual forms of apparatus, determining the carbon dioxid evolved either by increase of weight of potash bulbs or loss of weight of the apparatus. The former is preferable.

(f) DETERMINATION OF CHLORIN.

Determine as silver chlorid, either gravimetrically or by one of the standard volumetric processes, in a nitric acid or aqueous solution of the ash. Nitric acid may be used in (e) and the solution taken for this purpose.

VI.—METHODS FOR THE DETERMINATION OF CARBOHYDRATES IN AGRICULTURAL PRODUCTS, INCLUDING METHODS FOR THE DETERMINATION OF WATER AND ASH IN COMMERCIAL AND INDUSTRIAL SACCHARINE PRODUCTS.

1. Determination of Water.

(a) BY DRYING.

(1) IN SUGARS.—Dry from 2 to 5 grams in a flat dish (nickel, platinum, or aluminum), at the temperature of boiling water, for ten hours; cool in a desiccator and weigh; return to the oven and dry for an hour. If on weighing there be only a slight change of weight, the process may be considered finished; otherwise the drying must be continued until the loss of water in one hour is not greater than 0.20 per cent.

(2) IN MASSECUITES, MOLASSES, HONEYS, AND OTHER LIQUID AND SEMILIQUID PRODUCTS (PROVISIONAL METHOD).—Prepare pumice stone in two grades of fineness. One of these should pass through a 1 mm sieve, while the other should be composed of particles too large for a millimeter sieve, but sufficiently small to pass through a sieve having meshes 6 mm in diameter. Make the determination in flat metallic dishes or in shallow, flat-bottomed weighing bottles. Place a layer of the fine pumice stone 3 mm in thickness over the bottom of the dish, and upon this place a layer of the coarse pumice stone from 6 to 10 mm in thickness. Dry the dish thus prepared and weigh. Dilute the sample with a weighed portion of water in such a manner that the diluted material shall contain from 20 to 30 per cent of dry matter. Weigh into the dish, prepared as described above, such a quantity of the diluted sample as will yield, approximately, 1 gram of dry matter. Use a weighing bottle provided with a cork through which a pipette passes if this weighing can not be made with extreme rapidity. Place the dish in a water oven and dry to constant weight at the temperature of boiling water, making trial weighings at intervals of two hours. In case of materials containing much levulose or other readily decomposable substances, conduct the drying in vacuo at a lower temperature. In the case of very unstable material, the temperature can safely be lowered to 70°.

(b) FROM THE DENSITY.

(1) DETERMINATION OF SPECIFIC GRAVITY, WATER, AND TOTAL SOLIDS BY MEANS OF A SPINDLE.—The density of juices, sirups, etc., is most conveniently determined by means of Baumé's or Brix's hydrometer or areometer, preferably with the latter, as the graduations of the scale give close approximations to the percentages of total solids. The Brix spindle should be graduated to tenths. It is therefore desirable, for accuracy, that the range of degrees recorded by each individual spindle be as limited as possible, this end being best secured by the employment of sets, consisting of not less than three spindles. The solutions should be as nearly as possible of the same temperature as the air at the time of reading, and if the variation from the temperature of the graduation of the spindle amounts to more than 1°, compensation therefor must be made by reference to the table of corrections for temperature, page 58. This temperature should be 17.5°. Before taking the density of a juice, it should be allowed to stand in the cylinder until all air bubbles have escaped.

In case the sample is too dense to determine the density directly, a weighed portion of it must first be diluted with a weighed quantity of water, or a weighed portion must be dissolved and diluted to a known volume with water. In the first instance, the per cent of total solids is calculated by the following formula:

$$\text{Per cent of solids in the undiluted material} = \frac{WS}{w}.$$

S = per cent of solids in the diluted material.
W = weight of the diluted material.
w = weight of the sample taken for dilution.

When the dilution is made to a definite volume, the following formula is to be used:

$$\text{Per cent of solids in the undiluted material} = \frac{VDS}{W}.$$

V = volume of the diluted solution.
D = specific gravity of the diluted solution.
S = per cent of solids in the diluted solution.
W = weight of the sample taken for dilution.

A table for the comparison of specific gravities $\left(\frac{17.5^\circ}{17.5^\circ}\right)$, degrees Brix (per cent by weight of sucrose) and degrees Baumé, is given below.

(2) DETERMINATION OF SPECIFIC GRAVITY, WATER, AND TOTAL SOLIDS BY MEANS OF A PYCNOMETER.—When a more accurate determination of the per cent of solids or of water or of the specific gravity is desired, the determination should be made with a specific-gravity bottle or pycnometer. When of too high density for a direct determination, the sample may be diluted, as described under (1).

A table for the comparison of specific gravities, degrees Brix and degrees Baumé.[1]

Degree Brix or per cent by weight of sucrose.	Specific gravity.	Degree Baumé.	Degree Brix or per cent by weight of sucrose.	Specific gravity.	Degree Baumé.	Degree Brix or per cent by weight of sucrose.	Specific gravity.	Degree Baumé.
1.0	1.00388	0 6	33.0	1.14423	18.5	65.0	1.31989	35.6
2.0	1.00779	1.1	34 0	1.14915	19.05	66.0	1.32601	36.1
3 0	1.01173	1.7	35.0	1.15411	19.6	67.0	1.33217	36.6
4 0	1.01570	2 3	36.0	1.15911	20.1	68.0	1.33836	37.1
5.0	1.01970	2 8	37.0	1.16413	20.7	69.0	1.34460	37.6
6.0	1 02373	3.4	38.0	1.16920	21.2	70.0	1.35088	38.1
7.0	1.02779	4.0	39.0	1.17430	21.8	71.0	1.35720	38.6
8 0	1.03187	4.5	40.0	1.17943	22.3	72.0	1.36355	39.1
9.0	1.03599	5.1	41.0	1.18460	22.9	73.0	1.36995	39.6
10 0	1.04014	5.7	42.0	1.18981	23.4	74.0	1.37639	40.1
11.0	1 04431	6.2	43.0	1.19505	23.95	75.0	1.38287	40.6
12.0	1 04852	6.8	44.0	1.20033	24.5	76.0	1.38939	41.1
13.0	1.05276	7.4	45.0	1.20565	25.0	77.0	1.39595	41.6
14 0	1 05703	7.9	46.0	1.21100	25.6	78.0	1.40254	42.1
15.0	1.06133	8.5	47.0	1.21639	26.1	79.0	1.40918	42.6
16.0	1.06566	9 0	48.0	1.22182	26.6	80.0	1.41586	43.1
17.0	1.07002	9 6	49.0	1.22728	27.2	81.0	1.42258	43.6
18.0	1.07441	10.1	50.0	1.23278	27.7	82.0	1.42934	44.1
19.0	1.07884	10.7	51.0	1.23832	28.2	83.0	1.43614	44.6
20.0	1.08329	11.3	52.0	1.24390	28.8	84.0	1.44298	45.1
21.0	1.08778	11.8	53.0	1.24951	29.3	85.0	1.44986	45.5
22.0	1.09231	12.4	54.0	1.25517	29.8	86.0	1.45678	46.0
23.0	1.09686	13.0	55.0	1.26086	30.4	87.0	1.46374	46.5
24.0	1.10145	13.5	56.0	1.26658	30.9	88.0	1.47074	47.0
25.0	1.10607	14.1	57.0	1.27235	31.4	89.0	1.47778	47.45
26 0	1.11072	14.6	58.0	1.27816	31.9	90.0	1.48486	47.9
27.0	1.11541	15.2	59.0	1.28400	32.5	91.0	1.49199	48.5
28.0	1.12013	15.7	60.0	1.28989	33.0	92.0	1.49915	48.9
29.0	1.12488	16.3	61.0	1.29581	33.5	93.0	1.50635	49.4
30.0	1.12967	16.8	62.0	1.30177	34.0	94.0	1.51359	49.8
31.0	1.13449	17.4	63.0	1.30777	34.5	95.0	1.52087	50.3
32.0	1.13934	17.95	64.0	1.31381	35.1			

[1] Abridged from Frühling und Schulz, Anleitung zur Untersuchung der für die Zucker-Industrie in Betracht kommenden Rohmaterialien, etc., 1891, S. 57.

When the number expressing the specific gravity found by analysis falls between the numbers given in the above table, the exact equivalent in degrees Brix or Baumé is found by a simple calculation.

Example.—The pycnometer shows the specific gravity of a certain sirup to be 1.20909. The table shows that the corresponding degree Brix is between 45.0 and 46.0. Subtracting the specific gravity of a solution of 45° Brix from the corresponding figure for 46°, we have (expressing the specific gravities as whole numbers) 121,100 − 120,565 = 535, the difference in specific gravity for 1° Brix at this point in the table. Subtracting the specific gravity corresponding to 45° from the specific gravity found by analysis, we have 120,909 − 120,565 = 344. $\frac{344}{535} = 0.64$, the fraction of 1° Brix more than 45°. The degree Brix corresponding to a specific gravity of 1.20909 is therefore 45.64.

If the spindle reading or pycnometer determination is made at any other temperature than 17.5°, the result should be corrected by the use of the following table:

Table for correction of the readings of the Brix spindle when the reading is made at other than the standard temperature, 17.5°.[1]

[For temperatures below 17.5° the correction is to be subtracted.]

Temperature.	Degree Brix of the solution.												
	0	5	10	15	20	25	30	35	40	50	60	70	75
° C.													
0	0.17	0.30	0.41	0.52	0.62	0.72	0.82	0.92	0.98	1.11	1.22	1.25	1.29
5	0.23	0.30	0.37	0.44	0.52	0.59	0.65	0.72	0.75	0.80	0.88	0.91	0.94
10	0.20	0.26	0.29	0.33	0.36	0.39	0.42	0.45	0.48	0.50	0.54	0.58	0.61
11	0.18	0.23	0.26	0.28	0.31	0.34	0.36	0.39	0.41	0.43	0.47	0.50	0.53
12	0.16	0.20	0.22	0.24	0.26	0.29	0.31	0.33	0.34	0.36	0.40	0.42	0.46
13	0.14	0.18	0.19	0.21	0.22	0.24	0.26	0.27	0.28	0.29	0.33	0.35	0.39
14	0.12	0.15	0.16	0.17	0.18	0.19	0.21	0.22	0.22	0.23	0.26	0.28	0.32
15	0.09	0.11	0.12	0.14	0.14	0.15	0.16	0.17	0.16	0.17	0.19	0.21	0.25
16	0.06	0.07	0.08	0.09	0.10	0.10	0.11	0.12	0.12	0.12	0.14	0.16	0.18
17	0.02	0.02	0.03	0.03	0.03	0.04	0.04	0.04	0.04	0.04	0.05	0.05	0.06

[For temperatures above 17.5° the correction is to be added.]

18	0.02	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.02
19	0.06	0.08	0.08	0.09	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.08	0.06
20	0.11	0.14	0.15	0.17	0.17	0.18	0.18	0.18	0.19	0.19	0.18	0.15	0.11
21	0.16	0.20	0.22	0.24	0.24	0.25	0.25	0.25	0.26	0.26	0.25	0.22	0.18
22	0.21	0.26	0.29	0.31	0.31	0.32	0.32	0.32	0.33	0.34	0.32	0.29	0.25
23	0.27	0.32	0.35	0.37	0.38	0.39	0.39	0.39	0.40	0.42	0.39	0.36	$0.^{3}3$
24	0.32	0.38	0.41	0.43	0.44	0.46	0.46	0.47	0.47	0.50	0.46	0.43	$0.^{4}0$
25	0.37	0.44	0.47	0.49	0.51	0.53	0.54	0.55	0.55	0.58	0.54	0.51	$0.^{4}8$
26	0.43	0.50	0.54	0.56	0.58	0.60	0.61	0.62	0.62	0.66	0.62	0.58	$0.^{5}5$
27	0.49	0.57	0.61	0.63	0.65	0.68	0.68	0.69	0.70	0.74	0.70	0.65	$0.^{6}2$
28	0.56	0.64	0.68	0.70	0.72	0.76	0.76	0.78	0.78	0.82	0.78	0.72	$0.^{7}0$
29	0.63	0.71	0.75	0.78	0.79	0.84	0.84	0.86	0.86	0.90	0.86	0.80	$0.^{7}8$
30	0.70	0.78	0.82	0.87	0.87	0.92	0.92	0.94	0.94	0.98	0.94	0.88	$0.^{8}6$
35	1.10	1.17	1.22	1.24	1.30	1.32	1.33	1.35	1.36	1.39	1.34	1.27	$1.^{2}5$
40	1.50	1.61	1.67	1.71	1.73	1.79	1.79	1.80	1.82	1.83	1.78	1.69	$1.^{6}5$
50	.....	2.65	2.71	2.74	2.78	2.80	2.80	2.80	2.80	2.79	2.70	2.56	$2.^{5}1$
60	.....	3.87	3.88	3.88	3.88	3.88	3.88	3.88	3.90	3.82	3.70	3.43	$3.^{4}1$
70	.....	5.17	5.18	5.20	5.14	5.13	5.10	5.08	5.06	4.90	4.72	4.47	4.35
80	.....	.....	6.62	6.59	6.54	6.46	6.38	6.30	6.26	6.06	5.82	5.50	5.33
90	.....	.....	8.26	8.16	8.06	7.97	7.83	7.71	7.58	7.30	6.96	6.58	6.37
100	.....	.....	10.01	9.87	9.72	9.56	9.39	9.21	9.03	8.64	8.22	7.76	7.42

[1] Frühling und Schulz, Anleitung zur Untersuchung der für die Zucker-Industrie in Betracht kommenden Rohmaterialien, etc., 1891, S. 45.

Example.—A certain sugar solution shows a reading of 30.2° Brix at 30° C. To find the necessary correction for the conversion of this reading to the reading which would have been obtained if the observation had been made at 17.5° C., find the vertical column in the table headed 30° Brix, which is the nearest to the observed reading. Follow down this column until the number is reached which is opposite to the temperature of observation—in this case 30° C. The number found, 0.92, is to be added to the observed reading, making it 31.12.

2. Ash.

(a) determination of ash.

(1) Heat from 5 to 10 grams of the material (sugar, molasses, honey) in a platinum dish[1] of from 50 to 100 cc capacity at 100° until the water is expelled, and then slowly over a flame until intumescence ceases. The dish is then placed in a muffle and heated at low redness until a white ash is obtained.

For soluble ash digest the ash obtained as above with water, filter through a Gooch crucible, wash with hot water, dry the residue at 100°, and weigh. The difference of weights equals soluble ash.

(2) Use 50 mg of zinc oxid to 25 grams of molasses or 50 grams of sugar. Incorporate thoroughly by adding dilute alcohol and mixing; dry and ignite as above. Deduct the weight of zinc oxid used from the weight of ash.

(3) Carbonize the mass at a low heat, dissolve the soluble salts with hot water, burn the residual mass as above, add the solution of soluble salts, and evaporate to dryness at 100°; ignite gently, cool in a desiccator, and weigh.

(4) Saturate the sample with sulfuric acid, dry, ignite gently, then burn in a muffle at low redness. Deduct one-tenth of the weight of the ash, then calculate the per cent.

(5) Thoroughly mix 5 grams of the material with a somewhat larger weight of pure quartz sand in a platinum dish; ignite in a muffle at a moderate red heat.

(6) To avoid the correction of one-tenth, as proposed by Scheibler, and one-fifth, as proposed by Girard and Violette, when sugars are burned with sulfuric acid, Boyer suggests incineration with benzoic acid as giving the real quantity of mineral matter without correction.

The benzoic acid is dissolved in alcohol of 90 per cent, 25 grams of the acid to 100 cc of alcohol. Five grams of the sugar are weighed in a capsule and moistened with 1 cc of water. The capsule is heated slowly in order to caramelize the sugar without carbonizing it; 2 cc of the benzoic acid solution are next added, and the capsule warmed until all the alcohol is evaporated; the temperature is then raised until the sugar is converted into carbon. The decomposing benzoic acid produces abundant vapors, which render the mass extremely porous, especially if a circular motion is imparted to the capsule. The slow heating is continued until all the benzoic acid is volatilized. The carbon obtained is voluminous and of a brilliant black color. The incineration is accomplished in a muffle at a low red heat. The capsule should be weighed quickly when taken from the desiccator, in order to avoid the absorption of water by the alkaline carbonates. Ammonium benzoate may be employed instead of benzoic acid, and the analyst should previously assure himself that neither the acid nor the ammonium salt leaves a residue on incineration. In addition to giving the mineral matter directly, this method permits the determination of its composition also—a matter of no small importance.

(b) quantitative analysis of the ash.

Dissolve 10 grams of sugar in hot water and filter to remove sand, etc.; evaporate the filtrate and washings to dryness; carefully carbonize and extract with hot water until there is no longer any reaction for chlorin. Dry, ignite the residue, weigh as insoluble ash. Add the soluble portion, treat with hydrochloric acid in slight excess, and evaporate to dryness. Heat until all free hydrochloric acid is driven off, take up with water and a little hydrochloric acid, filter, and wash. The residue is silica. Add ammonia to the filtrate, boil, filter, and wash the residue, which is ferric

[1] If the substance contain tin or any other metal capable of uniting with platinum, a dish made of some other material must be used.

oxid and alumina. To the filtrate add ammonium oxalate, evaporate to dryness, ignite; moisten with ammonium carbonate; heat to low redness to expel ammonium salts, dissolve in water, filter, and wash. The residue on the filter represents calcium and magnesium carbonates. Evaporate the filtrate to a small bulk, add ammonium carbonate, and evaporate to dryness; drive off the excess of ammonium salts cautiously and weigh. This gives the alkalies as carbonates, and this weight added to the insoluble ash, formerly determined, gives the total carbonate ash.

3. Determination of Nitrogen.

Any of the methods adopted by the association for the estimation of nitrogen may be used (p. 15).

4. Determination of Reducing Sugars.

(a) preparation of reagents (soxhlet's modification of fehling's solution).

(1) 34.639 grams of $CuSO_4.5H_2O$ are dissolved in water and diluted to 500 cc.

(2) 173 grams of Rochelle salts and 50 grams of sodium hydroxid, dissolved in water and diluted to 500 cc. The requisite quantity of sodium hydroxid is best obtained by using 100 cc of a solution containing 500 grams of caustic soda in 1 liter. A solution of this strength has a specific gravity of 1.393 at 15°.

(3) Mix equal volumes of solutions (1) and (2) and boil immediately before use. The mixture is the mixed copper reagent to be used for all the methods given below except Allihn's method for dextrose, for which a special reagent must be used.

(b) volumetric methods.

(1) *Approximate volumetric method for rapid work.*

Place 10 cc of the mixed copper reagent in a large test tube and add 10 cc of distilled water. Heat to boiling, and gradually add small portions of the solution of the material to be tested, until the copper has been completely precipitated, boiling to complete the reaction after each addition. Two minutes' boiling is required for complete precipitation when the full amount of sugar solution has been added in one portion. When the end reaction is nearly reached and the amount of sugar solution to be added can no longer be judged by the color of the solution, a small portion of the liquid is removed by means of Knorr's modification of Wiley's filtering tube (any other rapid means of filtration may be used that removes but a small portion of the hot liquid), is transferred to a small porcelain crucible or test plate, acidified with dilute acetic acid, and tested for copper with a dilute solution of potassium ferrocyanid. The sugar solution should be of such a strength as will give a burette reading of 15 to 20 cc, and the number of successive additions should be as small as possible.

Since the factor for calculation varies with the minute details of manipulation, every operator must determine a factor for himself, using a known solution of a pure sample of the sugar that he desires to determine and keeping the conditions the same as those used for the determinations. For the standardization of a solution for the determination of invert sugar in sugar-house products, dissolve 2.5 grams of pure sucrose in 75 cc of water, add 5 cc of hydrochloric acid (specific gravity 1.188 at 15°), and invert according to method (b) under Optical Methods for the Determination of Sucrose by Inversion, page 58. Neutralize the acid with sodium carbonate and dilute to 1 liter. The 2.5 grams of sucrose become 2.6316 grams of invert sugar. The weight of invert sugar equivalent to 10 cc of the copper reagent is calculated as follows:

$$\frac{2.6316 \times \text{number of cc of the standard sugar solution used}}{1000} = X,$$

the weight of invert sugar required to completely precipitate the copper in 10 cc of

the reagent under the conditions used for the test titration. For the calculation of the result of the titration of an unknown solution:

Let X = the factor obtained as above;

V = the number of cc of unknown sugar solution required to precipitate the copper from 10 cc of copper solution;

W = the weight of the material under examination in 1 cc of the solution.

Then $\frac{100X}{VW}$ = per cent of invert sugar in the sample.

The calculation can be much simplified by so standardizing the copper reagent that 50 mg of invert sugar will be required to reduce the copper from 10 cc of the copper reagent. The various tables given in works on sugar analysis then become applicable. These tables are arranged for a "glucose normal solution" containing 5 grams of the material to be examined in 100 cc. When the weight per 100 cc is more or less than 5 grams, the number found in the table is increased or diminished accordingly.

(2) *Soxhlet's exact method.*

A preliminary titration is made to determine the approximate percentage of reducing sugar in the material under examination. A solution is prepared which contains approximately 1 per cent of reducing sugar. Place in a beaker 100 cc of the mixed copper reagent and approximately the amount of the sugar solution for its complete reduction. Boil for two minutes. Filter through a folded filter and test a portion of the filtrate for copper by use of acetic acid and potassium ferrocyanid. Repeat the test, varying the volume of sugar solution, until two successive amounts of sugar solution are found which differ by 0.1 cc, one giving complete reduction and the other leaving a small amount of copper in solution. The mean of these two readings is taken as the volume of the solution required for the complete precipitation of 100 cc of the copper reagent.

Under these conditions 100 cc of the mixed copper reagent require 0.475 gram of anhydrous dextrose, or 0.494 gram of invert sugar, for complete reduction. The percentage is calculated by the following formula:

W = the weight of the sample in 1 cc of the sugar solution;

V = the volume of the sugar solution required for the complete reduction of 100 cc of the copper reagent.

Then $\frac{100 \times 0.475}{VW}$ = per cent of dextrose,

or $\frac{100 \times 0.494}{VW}$ = per cent of invert sugar.

(3) *Volumetric method for the determination of the copper reduced when the manipulations are made to accord with any one of the various gravimetric methods.* ((*e*), p. 56.)

(c) GRAVIMETRIC METHODS.

(1) *Methods for conducting the boiling with copper solution.*

(*a*) METHOD FOR MATERIALS CONTAINING 1 PER CENT OR LESS OF INVERT SUGAR AND A HIGH PERCENTAGE OF SUCROSE.—The solution of the material to be examined is so prepared as to contain 20 grams in 100 cc, and it must be freed from suspended impurities by filtration through paper or by use of lead acetate, removing the excess of lead by means of sodium carbonate. In a beaker of 250 cc capacity place 50 cc of the mixed copper reagent and 50 cc of the sugar solution. Heat this mixture at such a rate that approximately four minutes are required to bring it to the boiling point, and boil for exactly two minutes. Add 100 cc of cold, recently boiled, distilled water. Filter immediately and finish the determination by one of the methods given below (p. 55) for the determination of the copper contained in the precipitate of cuprous oxid obtained in the determination of reducing sugars.

The corresponding percentage of invert sugar is found by the use of the following table:

Herzfeld's table for the determination of invert sugar in materials containing 1 per cent or less of invert sugar and a high percentage of sucrose.

Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.	Copper reduced by 10 grams of material.	Invert sugar.
Milligrams.	*Per cent.*	*Milligrams.*	*Per cent.*	*Milligrams.*	*Per cent.*
50	0.05	120	0.40	190	0.79
55	0.07	125	0.43	195	0.82
60	0.09	130	0.45	200	0.85
65	0.11	135	0.48	205	0.88
70	0.14	140	0.51	210	0.90
75	0.16	145	0.53	215	0.93
80	0.19	150	0.56	220	0.96
85	0.21	155	0.59	225	0.99
90	0.24	160	0.62	230	1.02
95	0.27	165	0.65	235	1.05
100	0.30	170	0.68	240	1.07
105	0.32	175	0.71	245	1.10
110	0.35	180	0.74		
115	0.38	185	0.76		

(*b*) METHOD FOR MATERIALS CONTAINING MORE THAN 1 PER CENT OF INVERT SUGAR.—Prepare a solution of the material to be examined in such a manner that it contains 20 grams in 100 cc after clarification and the removal of the excess of lead. Prepare a series of solutions in large test tubes by adding 1, 2, 3, 4, 5, etc., cc of this solution to each successively. Add 5 cc of the mixed copper reagent to each, heat to boiling, boil two minutes, and filter. Note the volume of sugar solution which gives the filtrate lightest in tint, but still distinctly blue. Place twenty times this volume of the sugar solution in a 100 cc flask, dilute to the mark, and mix well. Use 50 cc of the solution for the determination, which is conducted as described under (*a*) until the weight of copper is obtained. For the calculation of the result use the following formulas and table of factors of Meissl and Hiller:

Let Cu = the weight of copper obtained;

P = the polarization of the sample;

W = the weight of the sample in the 50 cc of the solution used for determination;

F = the factor obtained from the table for conversion of copper to invert sugar;

$\frac{Cu}{2}$ = approximate absolute weight of invert sugar = Z;

$Z \times \frac{100}{W}$ = approximate per cent of invert sugar = y;

$\frac{100\ P}{P+y}$ = R, relative number for sucrose;

100 − R = I, relative number for invert sugar;

$\frac{Cu\ F}{W}$ = per cent of invert sugar.

Z facilitates reading the vertical columns; and the ratio of R to I, the horizontal columns of the table, for the purpose of finding the factor (F) for calculation of copper to invert sugar.

Example.—The polarization of a sugar is 86.4, and 3.256 grams of it (W) are equivalent to 0.290 gram of copper. Then:

$$\frac{Cu}{2}=\frac{.290}{2}=0.145=Z$$

$$Z\times\frac{100}{W}=0.145\times\frac{100}{3.256}=4.45=Y$$

$$\frac{100\ P}{P+y}=\frac{8640}{86.4+4.45}=95.1=R$$

$$100-R=100-95.1=I=4.9$$

$$R:I=95.1:4.9$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to Z, 145, and the horizontal column headed 95:5 is nearest to the ratio of R to I, 95.1:4.9. Where these columns meet we find the factor 51.2, which enters into the final calculation:

$$\frac{Cu\ F}{W}=\frac{0.290\times51.2}{3.256}=4.56\text{ per cent of invert sugar.}$$

Meissl and Hiller's factors for the determination of more than 1 per cent of invert sugar.

Ratio of sucrose to invert sugar= R:I.	Approximate absolute weight of invert sugar = Z.						
	200 milligrams.	175 milligrams.	150 milligrams.	125 milligrams.	100 milligrams.	75 milligrams.	50 milligrams.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
0:100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10:90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20:80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30:70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40:60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50:50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60:40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70:30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80:20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90:10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91:9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92:8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93:7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94:6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95:5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96:4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97:3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98:2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99:1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

(*c*) Allihn's method for the determination of dextrose.

Reagents:

34.639 grams of $CuSO_4.5H_2O$, dissolved in water and diluted to 500 cc.

173 grams of Rochelle salts
125 grams of potassium hydroxid } dissolved in water and diluted to 500 cc.

Place 30 cc of the copper solution, 30 cc of the alkaline tartrate solution, and 60 cc of water in a beaker, and heat to boiling. Add 25 cc of the solution of the material to be examined, which must be so prepared as not to contain more than 1 per cent of dextrose, and boil for two minutes. Filter immediately without diluting

and obtain the weight of copper by one of the methods given on page 55. The corresponding weight of dextrose is found by the following table:

Allihn's table for the determination of dextrose.[1]

Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
10	6.1	58	29.8	106	54.0	154	78.6	202	103.7
11	6.6	59	30.3	107	54.5	155	79.1	203	104.2
12	7.1	60	30.8	108	55.0	156	79.6	204	104.7
13	7.6	61	31.3	109	55.5	157	80.1	205	105.3
14	8.1	62	31.8	110	56.0	158	80.7	206	105.8
15	8.6	63	32.3	111	56.5	159	81.2	207	106.3
16	9.0	64	32.8	112	57.0	160	81.7	208	106.8
17	9.5	65	33.3	113	57.5	161	82.2	209	107.4
18	10.0	66	33.8	114	58.0	162	82.7	210	107.9
19	10.5	67	34.3	115	58.6	163	83.3	211	108.4
20	11.0	68	34.8	116	59.1	164	83.8	212	109.0
21	11.5	69	35.3	117	59.6	165	84.3	213	109.5
22	12.0	70	35.8	118	60.1	166	84.8	214	110.0
23	12.5	71	36.3	119	60.6	167	85.3	215	110.6
24	13.0	72	36.8	120	61.1	168	85.9	216	111.1
25	13.5	73	37.3	121	61.6	169	86.4	217	111.6
26	14.0	74	37.8	122	62.1	170	86.9	218	112.1
27	14.5	75	38.3	123	62.6	171	87.4	219	112.7
28	15.0	76	38.8	124	63.1	172	87.9	220	113.2
29	15.5	77	39.3	125	63.7	173	88.5	221	113.7
30	16.0	78	39.8	126	64.2	174	89.0	222	114.3
31	16.5	79	40.3	127	64.7	175	89.5	223	114.8
32	17.0	80	40.8	128	65.2	176	90.0	224	115.3
33	17.5	81	41.3	129	65.7	177	90.5	225	115.9
34	18.0	82	41.8	130	66.2	178	91.1	226	116.4
35	18.5	83	42.3	131	66.7	179	91.6	227	116.9
36	18.9	84	42.8	132	67.2	180	92.1	228	117.4
37	19.4	85	43.4	133	67.7	181	92.6	229	118.0
38	19.9	86	43.9	134	68.2	182	93.1	230	118.5
39	20.4	87	44.4	135	68.8	183	93.7	231	119.0
40	20.9	88	44.9	136	69.3	184	94.2	232	119.6
41	21.4	89	45.4	137	69.8	185	94.7	233	120.1
42	21.9	90	45.9	138	70.3	186	95.2	234	120.7
43	22.4	91	46.4	139	70.8	187	95.7	235	121.2
44	22.9	92	46.9	140	71.3	188	96.3	236	121.7
45	23.4	93	47.4	141	71.8	189	96.8	237	122.3
46	23.9	94	47.9	142	72.3	190	97.3	238	122.8
47	24.4	95	48.4	143	72.9	191	97.8	239	123.4
48	24.9	96	48.9	144	73.4	192	98.4	240	123.9
49	25.4	97	49.4	145	73.9	193	98.9	241	124.4
50	25.9	98	49.9	146	74.4	194	99.4	242	125.0
51	26.4	99	50.4	147	74.9	195	100.0	243	125.5
52	26.9	100	50.9	148	75.5	196	100.5	244	126.0
53	27.4	101	51.4	149	76.0	197	101.0	245	126.6
54	27.9	102	51.9	150	76.5	198	101.5	246	127.1
55	28.4	103	52.4	151	77.0	199	102.0	247	127.6
56	28.8	104	52.9	152	77.5	200	102.6	248	128 1
57	29.3	105	53.5	153	78.1	201	103.1	249	128.7

[1] J. prakt. Chem., 1880, **22**, 46.

Allihn's table for the determination of dextrose—Continued.

Milligrams of copper	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.	Milligrams of copper.	Milligrams of dextrose.
250	129.2	293	152.7	336	176.5	379	200.8	422	225.7
251	129.7	294	153.2	337	177.0	380	201.4	423	226.3
252	130.3	295	153.8	338	177.6	381	202.0	424	226.9
253	130.8	296	154.3	339	178.1	382	202.5	425	227.5
254	131.4	297	154.9	340	178.7	383	203.1	426	228.0
255	131.9	298	155.4	341	179.3	384	203.7	427	228.6
256	132.4	299	156.0	342	179.8	385	204.3	428	229.2
257	133.0	300	156.5	343	180.4	386	204.8	429	229.8
258	133.5	301	157.1	344	180.9	387	205.4	430	230.4
259	134.1	302	157.6	345	181.5	388	206.0	431	231.0
260	134.6	303	158.2	346	182.1	389	206.5	432	231.6
261	135.1	304	158.7	347	182.6	390	207.1	433	232.2
262	135.7	305	159.3	348	183.2	391	207.7	434	232.8
263	136.2	306	159.8	349	183.7	392	208.3	435	233.4
264	136.8	307	160.4	350	184.3	393	208.8	436	233.9
265	137.3	308	160.9	351	184.9	394	209.4	437	234.5
266	137.8	309	161.5	352	185.4	395	210.0	438	235.1
267	138.4	310	162.0	353	186.0	396	210.6	439	235.7
268	138.9	311	162.6	354	186.6	397	211.2	440	236.3
269	139.5	312	163.1	355	187.2	398	211.7	441	236.9
270	140.0	313	163.7	356	187.7	399	212.3	442	237.5
271	140.6	314	164.2	357	188.3	400	212.9	443	238.1
272	141.1	315	164.8	358	188.9	401	213.5	444	238.7
273	141.7	316	165.3	359	189.4	402	214.1	445	239.3
274	142.2	317	165.9	360	190.0	403	214.6	446	239.8
275	142.8	318	166.4	361	190.6	404	215.2	447	240.4
276	143.3	319	167.0	362	191.1	405	215.8	448	241.0
277	143.9	320	167.5	363	191.7	406	216.4	449	241.6
278	144.4	321	168.1	364	192.3	407	217.0	450	242.2
279	145.0	322	168.6	365	192.9	408	217.5	451	242.8
280	145.5	323	169.2	366	193.4	409	218.1	452	243.4
281	146.1	324	169.7	367	194.0	410	218.7	453	244.0
282	146.6	325	170.3	368	194.6	411	219.3	454	244.6
283	147.2	326	170.9	369	195.1	412	219.9	455	245.2
284	147.7	327	171.4	370	195.7	413	220.4	456	245.7
285	148.3	328	172.0	371	196.3	414	221.0	457	246.3
286	148.8	329	172.5	372	196.8	415	221.6	458	246.9
287	149.4	330	173.1	373	197.4	416	222.2	459	247.5
288	149.9	331	173.7	374	198.0	417	222.8	460	248.1
289	150.5	332	174.2	375	198.6	418	223.3	461	248.7
290	151.0	333	174.8	376	199.1	419	223.9	462	249.3
291	151.6	334	175.3	377	199.7	420	224.5	463	249.9
292	152.1	335	175.9	378	200.3	421	225.1		

(2) *Methods for the determination of the copper contained in the precipitate of cuprous oxid obtained in the determination of reducing sugars.*

(*a*) METHOD REQUIRING REDUCTION IN HYDROGEN.—Filter immediately through a weighed filtering tube of hard glass, under pressure. The asbestus film in the filtering tube is supported by a perforated disk or cone of platinum, and should be washed free from loose fibers before weighing, and moistened previous to the filtration. The tube is provided with a detachable funnel during the filtration, so that

none of the precipitate accumulates near the top, where it could be removed by the cork used during the reduction of the cuprous oxid. The precipitate is all transferred to the filter and thoroughly washed with hot water, following the water by alcohol and ether successively. After being dried, the tube is connected with an apparatus for supplying a continuous current of dry hydrogen, gently heated until the cuprous oxid is completely reduced to the metallic state, cooled in the current of hydrogen, and weighed.

(*b*) ELECTROLYTIC METHOD BY SOLUTION IN NITRIC ACID AND SUBSEQUENT EVAPORATION WITH EXCESS OF SULFURIC ACID.—The filtration, after reduction, is made in a Gooch crucible, and the beaker and precipitate thoroughly washed with hot water without any effort to transfer the precipitate to the filter. Wash the asbestus film and the adhering cuprous oxid into the beaker by means of hot dilute nitric acid. After the copper is all in solution, refilter through a Gooch crucible with a thin film of asbestus and wash thoroughly with hot water. Add 10 cc of dilute sulfuric acid (containing 200 cc of sulfuric acid—specific gravity 1.84—per liter) and evaporate the filtrate on the steam bath until the copper salt has largely crystallized out. Heat carefully on a hot plate or over a piece of asbestus board until the evolution of the white fumes shows that the excess of nitric acid is removed. Add from 8 to 10 drops of nitric acid (specific gravity, 1.42) and rinse into a platinum dish of from 100 to 125 cc capacity. Precipitate the copper by electrolysis. Wash thoroughly with water before breaking the current, remove the dish from the circuit, wash with alcohol and ether successively, dry at a temperature that can be borne by the hand, and weigh. If preferred, the electrolysis can be conducted in a beaker, the copper being deposited upon a weighed platinum cylinder.

(*c*) ELECTROLYTIC METHOD BY SOLUTION IN A MIXTURE OF SULFURIC AND NITRIC ACIDS.—The filtration and washing are conducted as under (*b*). Transfer the asbestus film from the crucible to the beaker by means of a glass rod, and rinse the crucible with about 30 cc of a boiling mixture of dilute sulfuric and nitric acids, containing 65 cc of sulfuric acid (specific gravity, 1.84) and 50 cc of nitric acid (specific gravity, 1.42) per liter. Heat and agitate until solution is complete; filter and electrolyze as under (*b*).

(*d*) ELECTROLYTIC METHOD BY SOLUTION IN NITRIC ACID.—Filter and wash as under (*b*). Transfer the asbestus film and adhering oxid to the beaker. Dissolve the oxid still remaining in the crucible by means of 2 cc of nitric acid (specific gravity, 1.42), adding it with a pipette and receiving the solution in the beaker containing the asbestus film. Rinse the crucible with a jet of water, allowing the rinsings to flow into the beaker. Heat the contents of the beaker until the copper is all in solution; filter out the asbestus, dilute the filtrate to a volume of 100 cc, or more, and electrolyze. According to Formanek, a solution of copper nitrate can be successfully electrolyzed if it contains as high as 4 per cent of nitric acid. When a nitrate solution is electrolyzed, the first washing of the deposit should be made with water acidulated with sulfuric acid, in order that the nitric acid may be all removed before the current is interrupted.

(*e*) VOLUMETRIC PERMANGANATE METHOD (ADOPTED PROVISIONALLY).—Filter and wash the cuprous oxid as described for method (*b*). Transfer the asbestus film to the beaker with a glass rod, add about 30 cc of hot water, and beat the precipitate and asbestus up thoroughly with the rod. Rinse the crucible with 50 cc of a hot saturated solution of ferric sulfate in 20 per cent sulfuric acid, receiving the rinsings in the beaker containing the precipitate. After the cuprous oxid is all dissolved, wash the solution into a large Erlenmeyer flask and immediately titrate with standard solution of potassium permanganate. One cc of the permanganate solution should equal 0.010 gram of copper. In order to determine the strength of this solution, make six or more determinations with the same sugar solution, titrating one-half of the precipitates obtained, and determining the copper in the others by electrolysis. The average weight of copper obtained by electrolysis, divided by the average number of cubic

centimeters of permanganate solution required for the titration, is equal to the weight of copper equivalent to 1 cubic centimeter of the standard permanganate solution. A solution standardized with iron or oxalic acid will give too low results.

5. Determination of Sucrose.

(a) optical methods.

(1) *Preparation of reagents.*

(*a*) Lead subacetate solution.—Boil an aqueous solution of lead acetate with an excess of lead oxid (PbO) for half an hour and make the filtered solution of a concentration of not less than 1.25 specific gravity. Solid lead subacetate may be substituted for the normal salt and oxid in the preparation of the solution.

(*b*) Alumina cream.—Prepare a cold saturated solution of alum in water and divide into two unequal portions. Add a slight excess of ammonium hydrate to the larger portion, and then add by degrees the remaining alum solution until a faintly acid reaction is secured.

(2) *Determination.*

(*a*) In sugars, massecuites, etc.—Place in a tared dish the normal weight for the instrument employed; wash into a 100 cc flask; add water until the volume is about 85 cc. When the crystals are all dissolved, add sufficient lead subacetate to throw down all precipitable matter. With molasses and massecuites add sufficient acetic acid to convert the subacetate into the neutral acetate. Make up to the mark, using a little ether spray to break bubbles; filter, throwing away the first 10 to 15 cc; place in the observation tube and polarize. If too dark to read, filter through dry, finely powdered bone black, rejecting the first 30 to 40 cc.

For adjusting the polariscope, graduating flasks, etc., the usual methods are followed.

It must be remembered that a change of 5° in the temperature of the solution between the time of filling the flask to the mark and the time of making the reading will cause a change of volume which will give rise to an error of approximately 0.1 per cent in the polarization of materials containing from 90 to 100 per cent of sucrose, and an error of approximately 0.05 per cent in the case of materials containing 50 per cent of sucrose.

(*b*) In juices, etc.—Transfer, by means of a pipette, to the tared sugar dish, the normal or multiple normal weight of the juice or sirup to be analyzed.

In the case of juices and thin sirups, the contents of the dish are at once washed into the 100 cc flask.

The solution in the flask is made up to about 90 cc, and the least amount of lead subacetate solution sufficient to decolorize the contents is added. The solution is made up to the 100 cc mark and thoroughly shaken before filtering. In case much foaming and bubbling take place, rendering gauging difficult, the addition of a few drops of ether is found advantageous. The solution is then poured upon a dry filter and the filtrate is collected in a dry beaker, the first portion to pass through being rejected. If it is found impossible to obtain a perfectly clear filtrate, it may be necessary to decolorize with dry powdered bone black, either mixed with the liquid itself or placed upon the filter, and rejecting again the first portions of the filtrate.

In the case of dark-colored products, the addition of alumina cream immediately after the lead subacetate is quite advantageous, the quantity of the former employed being in excess of that of the latter. For many sugars the use of alumina cream alone will produce a satisfactory clarification. Where double polarization is employed, the filtrate used for the direct reading should be acidified with acetic acid, in order to decompose the compound of levulose and lead which has been formed.

In the case of juices and other dilute materials, the weighing of the sample may be avoided by placing 100 cc of the material in a flask marked at 100 and at 110 cc,

treating with clarifying reagents, filling to the 110 cc mark, and completing the determination as described above. Schmitz's table may be used for the calculation of the result, or the following formula may be employed:

$$\text{Per cent of sucrose} = \frac{1.1\ RN}{VD}$$

R = the polariscopic reading in a 200 mm tube.
N = the normal weight of the instrument used.
V = the volume of the sample taken for the determination; and
D = the specific gravity of the sample.

(b) OPTICAL METHODS BY INVERSION.—FOR RAW SUGAR, MOLASSES, ETC.

(1) *Modified method of Clerget.*[1]

Make up the solution as above, and place 50 cc of the filtrate in a flask marked at 50 and 55 cc. Fill to the upper mark with pure fuming hydrochloric acid and mix well; place in water and heat until the thermometer, with the bulb as near the center of the sugar solution in the flask as possible, marks 68°, consuming about fifteen minutes in the heating; remove, cool quickly to room temperature, and polarize, noting the temperature. If the sample contained originally any invert sugar, the second polarization should be made at approximately the same temperature as the first. The percentage of sucrose is calculated by the following formula:

S = percentage of sucrose.
a = first polarization.
b = second polarization (usually to the left).
$a - b$ = the algebraic difference between the two polarizations.
t = temperature of observation.

Then

$$S = \frac{a - b}{144 - \frac{t}{2}}$$

(2) *The official German method.*

This method is conducted as follows: 26.048 grams of the sample are dissolved in a sugar flask and the solution made up to 100 cc; 50 cc of this solution are transferred by means of a pipette to a flask graduated at 50 and 55 cc, enough lead subacetate solution added for clarification, the volume made up to the 55 cc mark, and the liquid thoroughly shaken and filtered. The filtrate is then polarized, the reading being corrected for the extra 5 cc. The liquid adhering to the pipette is washed into the 100 cc flask containing the remaining 50 cc (13.024 grams), 5 cc of concentrated hydrochloric acid (38 per cent, specific gravity 1.188 at 15°) added, and the flask placed in a water bath, the temperature of which is 70°. The contents of the flask should reach a temperature of 67–70° in two or three minutes, when the temperature should be maintained within this limit for exactly five minutes, keeping the temperature as nearly 69° as possible. The flask is then cooled rapidly and the solution made up to 100 cc. If the liquid be colored, it is shaken with from one-half to 1 gram of bone black and filtered through a double filter. It is then polarized in a 200 mm tube, which is provided with a thermometer. As the rotatory power of invert sugar is much influenced by the temperature, this factor must be regarded. The reading is best taken between 18° and 22° and the temperature accurately determined. The reading must also be doubled on account of the dilution of the liquid.

To calculate per cent of sucrose (R), the two readings are added together, the sum (S) multiplied by 100 and divided by $142.4 - \frac{1}{2}t$ where t is the temperature at which

[1] The reporter for 1895 recommended that this method be stricken from the methods in 1896.

the invert reading was taken. If the temperature be exactly 20°, the result can be made more accurate by substituting 142.66 for 142.4, thus:

$$R = \frac{100\ S}{142.66 - \frac{2^0}{2}} = \frac{100\ S}{132.66} = 0.7538\ S.$$

If very much invert sugar be present, both the direct and indirect readings must be taken at the same temperature.

(3) *Method of Lindet.*

Place 50 cc of the filtrate as above in a flask marked at 100.5 cc; add 5 grams of powdered zinc (zinc dust), place in boiling water, and when at the boiling point add, little by little, 5 cc of strongest hydrochloric acid. After the acid has all been added, cool and make up to mark. Polarize in a 400 mm tube, or multiply the reading in a 200 mm tube by 2. Calculate the percentage of sucrose as before.

(c) GRAVIMETRIC METHOD.

Determine first any reducing sugar in the sample; then invert the sucrose according to (2) under Optical Methods by Inversion, neutralize the free acid, and redetermine the reducing sugar. Deduct the percentage of reducing sugar obtained at first, and the remainder will be the reducing sugar derived from the sucrose; multiply this number by 0.95 to obtain the percentage of sucrose in the sample.

6. DETERMINATION OF LACTOSE.

(a) OPTICAL METHOD FOR THE DETERMINATION OF LACTOSE IN MILK.

(1) *Preparation of reagents.*

(*a*) ACID MERCURIC NITRATE.—Dissolve mercury in double its weight of nitric acid, specific gravity 1.42. Add to the solution an equal volume of water. One cc of this reagent is sufficient for the quantities of milk mentioned below. Larger quantities may be used without affecting the results of polarization.

(*b*) MERCURIC IODID WITH ACETIC ACID.—33.2 grams of potassium iodid, 13.5 grams of mercuric chlorid, 20 cc of glacial acetic acid, and 640 cc of water.

(2) *Apparatus.*

One pipette or burette of 100 cc capacity, graduated in cubic centimeters and tenths of cubic centimeters; sugar flasks, marked at 102.4 cc when a polariscope is used for which the normal weight of sucrose is 16.19 grams, marked at 102.6 cc when the normal weight of sucrose for the polariscope used is 26.048 grams; filters, observation tubes, and polariscope; hydrometer and cylinder; thermometers.

(3) *Determination.*

The milk should be at a constant temperature and its specific gravity determined with a delicate hydrometer. Where greater accuracy is required, a pycnometer is used.

The quantities of the milk measured for polarization vary with the specific gravity of the milk as well as with the polariscope used. The quantity to be measured in any case will be found in the following table:

Specific gravity.	Volume of milk to be used.	
	For polariscopes of which the sucrose normal weight is 16.19 grams.	For polariscopes of which the sucrose normal weight is 26.048 grams.
	Cubic centimeters.	*Cubic centimeters.*
1.024	60.0	64.4
1.026	59.9	64.3
1.028	59.8	64.15
1.030	59.7	64.0
1.032	59.6	63.9
1.034	59.5	63.8
1.035	59.35	63.7

Place the quantity of milk, indicated in the table, in a flask graduated at 102.4, or 102.6 cc, according to the polariscope used. Add 1 cc of mercuric nitrate solution or 30 cc of mercuric iodid solution (an excess of these reagents does no harm), fill to the mark, agitate, filter through a dry filter, and polarize. It is not necessary to heat before polarizing. In case a 200 mm observation tube is used, the polariscopic reading is to be divided by 3 when the sucrose normal weight of the instrument employed is 16.19 grams, or by 2 when the normal weight of the instrument is 26.048 grams. When a 400 mm tube is used, these divisors become 6 and 4, respectively. For the calculation of the above table, the specific rotatory power of lactose is taken as 52.53°, and the corresponding number for sucrose as 66.5°. The lactose normal weight to read 100° on the sugar scale for instruments of the first class is 20.496 grams, and for instruments of the second class 32.975 grams.

(b) SOXHLET'S METHOD USING ALKALINE COPPER SOLUTION.

(1) *Preparation of the milk solution.*

Dilute 25 cc of the milk with 400 cc of water and add 10 cc of a solution of copper sulfate of the strength given for Soxhlet's modification of Fehling's solution, page 50; add about 7.5 cc of a solution of potassium hydroxid of such strength that one volume of it is just sufficient to completely precipitate the copper as suboxid from one volume of the solution of copper sulfate. In place of a solution of potassium hydroxid of this strength, 8.8 cc of a half normal solution of sodium hydroxid may be used. After the addition of the alkali solution the mixture must still have an acid reaction and contain copper in solution (p. 51). Fill the flask to the mark, agitate, and filter through a dry filter.

(2) *Determination.*

Place 50 cc of the mixed copper reagent in a beaker and heat to the boiling point. While boiling briskly add 100 cc of the milk solution, prepared as directed in the preceding paragraph, and boil for six minutes. Filter immediately and determine the amount of copper reduced by one of the methods given under Reducing Sugars, page 55. Obtain the weight of lactose equivalent to the weight of copper found from the following table:

Table for the determination of lactose.

[Calculated by E. Wein from data obtained by F. Soxhlet. Dr. Ernst Wein, Tabellen zur quantitativen Bestimmung der Zuckerarten, S. 9.]

Milligrams of copper.	Milligrams of lactose.	Milligrams of copper.	Milligrams of lactose.	Milligrams of copper	Milligrams of lactose.	Milligrams of copper.	Milligrams of lactose.	Milligrams of copper.	Milligrams of lactose.
100	71.6	151	109.6	202	148.5	253	187.1	304	227.5
101	72.4	152	110.3	203	149.2	254	187.9	305	228.3
102	73.1	153	111.1	204	150.0	255	188.7	306	229.1
103	73.8	154	111.9	205	150.7	256	189.4	307	229.8
104	74.6	155	112.6	206	151.5	257	190.2	308	230.6
105	75.3	156	113.4	207	152.2	258	191.0	309	231.4
106	76.1	157	114.1	208	153.0	259	191.8	310	232.2
107	76.8	158	114.9	209	153.7	260	192.5	311	232.9
108	77.6	159	115.6	210	154.5	261	193.3	312	233.7
109	78.3	160	116.4	211	155.2	262	194.1	313	234.5
110	79.0	161	117.1	212	156.0	263	194.9	314	235.3
111	79.8	162	117.9	213	156.7	264	195.7	315	236.1
112	80.5	163	118.6	214	157.5	265	196.4	316	236.8
113	81.3	164	119.4	215	158.2	266	197.2	317	237.6
114	82.0	165	120.2	216	159.0	267	198.0	318	238.4
115	82.7	166	120.9	217	159.7	268	198.8	319	239.2
116	83.5	167	121.7	218	160.4	269	199.5	320	240.0
117	84.2	168	122.4	219	161.2	270	200.3	321	240.7
118	85.0	169	123.2	220	161.9	271	201.1	322	241.5
119	85.7	170	123.9	221	162.7	272	201.9	323	242.3
120	86.4	171	124.7	222	163.4	273	202.7	324	243.1
121	87.2	172	125.5	223	164.2	274	203.5	325	243.9
122	87.9	173	126.2	224	164.9	275	204.3	326	244.6
123	88.7	174	127.0	225	165.7	276	205.1	327	245.4
124	89.4	175	127.8	226	166.4	277	205.9	328	246.2
125	90.1	176	128.5	227	167.2	278	206.7	329	247.0
126	90.9	177	129.3	228	167.9	279	207.5	330	247.7
127	91.6	178	130.1	229	168.6	280	208.3	331	248.5
128	92.4	179	130.8	230	169.4	281	209.1	332	249.2
129	93.1	180	131.6	231	170.1	282	209.9	333	250.0
130	93.8	181	132.4	232	170.9	283	210.7	334	250.8
131	94.6	182	133.1	233	171.6	284	211.5	335	251.6
132	95.3	183	133.9	234	172.4	285	212.3	336	252.5
133	96.1	184	134.7	235	173.1	286	213.1	337	253.3
134	96.9	185	135.4	236	173.9	287	213.9	338	254.1
135	97.6	186	136.2	237	174.6	288	214.7	339	254.9
136	98.3	187	137.0	238	175.4	289	215.5	340	255.7
137	99.1	188	137.7	239	176.2	290	216.3	341	256.5
138	99.8	189	138.5	240	176.9	291	217.1	342	257.4
139	100.5	190	139.3	241	177.7	292	217.9	343	258.2
140	101.3	191	140.0	242	178.5	293	218.7	344	259.0
141	102.0	192	140.8	243	179.3	294	219.5	345	259.8
142	102.8	193	141.6	244	180.1	295	220.3	346	260.6
143	103.5	194	142.3	245	180.8	296	221.1	347	261.4
144	104.3	195	143.1	246	181.6	297	221.9	348	262.3
145	105.1	196	143.9	247	182.4	298	222.7	349	263.1
146	105.8	197	144.6	248	183.2	299	223.5	350	263.9
147	106.6	198	145.4	249	184.0	300	224.4	351	264.7
148	107.3	199	146.2	250	184.8	301	225.2	352	265.5
149	108.1	200	146.9	251	185.5	302	225.9	353	266.3
150	108.8	201	147.7	252	186.3	303	226.7	354	267.2

Table for the determination of lactose—Continued.

[Calculated by E. Wein from data obtained by F. Soxhlet. Dr. Ernst Wein, Tabellen zur quantitativen Bestimmung der Zuckerarten, S. 9.]

Milligrams of copper.	Milligrams of lactose.	Milligrams of copper.	Milligrams of lactose.	Milligrams of copper.	Milligrams of lactose.	Milligrams of copper.	Milligrams of lactose.	Milligrams of copper	Milligrams of lactose.
355	268.0	364	275.3	373	283.1	382	290.8	391	298.5
356	268.8	365	276.2	374	283.9	383	291.7	392	299.4
357	269.6	366	277.1	375	284.8	384	292.5	393	300.3
358	270.4	367	277.9	376	285.7	385	293.4	394	301.1
359	271.2	368	278.8	377	286.5	386	294.2	395	302.0
360	272.1	369	279.6	378	287.4	387	295.1	396	302.8
361	272.9	370	280.5	379	288.2	388	296.0	397	303.7
362	273.7	371	281.4	380	289.1	389	296.8	398	304.6
363	274.5	372	282.2	381	289.9	390	297.7	399	305.4
								400	306.3

7. Provisional Methods for the Determination of Carbohydrates in Grains and By-Product Cattle Foods.

(a) Determination of reducing sugars (estimated as dextrose).

Stir 3 grams of the sample in a beaker with 50 cc of water for an hour. Filter into a quarter-liter flask, wash, and make up to mark. Determine reducing sugar as dextrose according to method (*c*), page 53. If the solution be difficult to filter, 2 cc of alumina cream should be added.

(b) Determination of sucrose.

Determine the sucrose in 50 cc of the filtrate obtained above according to method (*c*), page 53. Before calculating the invert sugar obtained as sucrose, deduct the reducing sugar determined as dextrose in (a).

(c) Determination of starch.[1]

Heat the insoluble residue obtained under (a) for 3 hours with 200 cc of water and 20 cc of hydrochloric acid (specific gravity 1.125) in a flask provided with a reflux condenser. Cool, and neutralize with sodium carbonate. Complete the volume to a quarter of a liter, filter, and determine the dextrose in an aliquot portion of the filtrate by Allihn's method. The weight of dextrose obtained multiplied by 0.9 will give the weight of starch.

(d) Determination of pentosans by the Tollens method with Krug's modification.

From 3 to 5 grams of the sample are placed in a flask with 100 cc of hydrochloric acid of 1.06 specific gravity. The mixture is distilled and the distillate collected in a graduated receiver. Each 10 cc lost by distillation is replaced by adding 10 cc of hydrochloric acid of the strength mentioned above. Continue the process as long as the distillate shows the furfurol reaction with anilin acetate.

Neutralize the distillate with sodium carbonate. If the volume be less than 400 cc, add 10.2 grams of sodium chlorid for each 50 cc less than that volume and complete the volume with water to 400 cc. Add 10 cc of a 12 per cent solution of phenylhydrazin in glacial acetic acid. Stir for half an hour with a stirring machine, and allow to stand for twenty-four hours. Filter on asbestus in a long glass tube and wash with 100 cc of cold water. Dissolve the precipitate on the filter with a small

[1] Note by Editor.—In this method there will be included as starch the pentosans and other carbohydrate bodies present which suffer hydrolysis on boiling with hydrochloric acid.

quantity of warm alcohol, collecting in a small tared flask. Evaporate the alcohol and dry at 60°.

From the weight of furfurol hydrazone the pentoses are obtained by the following factors:

Hydrazone × 0.9865 = xylose.
Hydrazone × 1.2126 = arabinose.
Hydrazone × 1.0995 = pentose.
Hydrazone × 0.7440 = pentosan.

(e) DETERMINATION OF CRUDE FIBER.

Extract 2 grams of the substance with ordinary ether, at least nearly completely, or use the residue from the determination of the ether extract. To this residue, in a 500 cc flask, add 200 cc of boiling 1.25 per cent sulfuric acid; connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once, and continue the boiling for thirty minutes. A blast of air conducted into the flask may serve to reduce the frothing of the liquid. Filter; wash thoroughly with boiling water till the washings are no longer acid; rinse the substance back into the same flask with 200 cc of a boiling 1.25 per cent solution of sodium hydroxid, nearly free from sodium carbonate; boil at once, and continue the boiling for thirty minutes in the same manner as directed above for the treatment with acid. Filter in a Gooch crucible, and wash with boiling water till the washings are neutral; dry at 110°; weigh; incinerate completely. The loss of weight is crude fiber.

The filter used for the first filtration may be linen, one of the forms of glass wool or asbestus filters, or any other form that secures clear and reasonably rapid filtration. The solutions of sulfuric acid and sodium hydroxid are to be made up of the specified strength, determined accurately by titration and not merely from specific gravity.

VII.—METHODS FOR THE ANALYSIS OF FERMENTED AND DISTILLE LIQUORS.

1. Determination of Specific Gravity.

The specific gravity is taken at 15.5° by means of a pycnometer or a Westphal balance controlled by a pycnometer, or a delicate hydrometer may be used which has been carefully calibrated by a pycnometer.

2. Determination of Alcohol.

(a) IN FERMENTED LIQUORS.

(1) *By weight.*

One hundred cc of the liquor are measured into a flask of from 250 to 300 cc capacity, 50 cc of water added, the flask attached to a vertical condenser by means of a bent tube, and 100 cc distilled. The specific gravity of the distillate is determined as in 1. The distillate is also weighed, or its weight calculated from the specific gravity. The corresponding percentage of alcohol by weight is obtained from the appended table, and this figure multiplied by the weight of the distillate, and the result divided by the weight of the sample taken, gives the per cent of alcohol by weight contained therein.

(2) *By volume.*

The percentage of alcohol by volume of the liquor is the same as that of the distillate, and is obtained from the appended table.

(b) IN DISTILLED LIQUORS.

(1) *By weight.*

About 30 grams of the liquor are weighed, diluted to 150 cc, 100 cc distilled, and the per cent of alcohol by weight determined as under Fermented Liquors.

(2) *By volume.*

The percentage of alcohol by volume in the distillate is obtained from the appended table, this figure divided by the volume of the liquor taken for the determination (calculated from the specific gravity), and the result multiplied by 100.

Percentage of alcohol by weight and by volume.

[Recalculated from the determinations of Gilpin, Drinkwater, and Squibb, by EDGAR RICHARDS.]

Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at $\frac{60}{60}$° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
1.00000	**0.00**	**0.00**	**0.99629**	**2.50**	**1.99**	**0.99281**	**5.00**	**4.00**	**0.98959**	**7.50**	**6.02**
0.99992	.05	.04	622	.55	2.03	274	.05	.04	953	.55	.06
984	.10	.08	615	.60	.07	268	.10	.08	947	.60	.10
976	.15	.12	607	.65	.11	261	.15	.12	940	.65	.14
968	.20	.16	600	.70	.15	255	.20	.16	934	.70	.18
961	.25	.20	593	.75	.19	248	.25	.20	928	.75	.22
953	.30	.24	586	.80	.23	241	.30	.24	922	.80	.26
945	.35	.28	579	.85	.27	235	.35	.28	916	.85	.30
937	.40	.32	571	.90	.31	228	.40	.32	909	.90	.34
930	.45	.36	564	.95	.35	222	.45	.36	903	.95	.38
.99923	**0.50**	**0.40**	**.99557**	**3.00**	**2.39**	**.99215**	**5.50**	**4.40**	**.98897**	**8.00**	**6.42**
915	.55	.44	550	.05	.43	208	.55	.44	891	.05	.46
907	.60	.48	543	.10	.47	202	.60	.48	885	.10	.50
900	.65	.52	536	.15	.51	195	.65	.52	879	.15	.54
892	.70	.56	529	.20	.55	189	.70	.56	873	.20	.58
884	.75	.60	522	.25	.59	182	.75	.60	867	.25	.62
877	.80	.64	515	.30	.64	175	.80	.64	861	.30	.67
869	.85	.67	508	.35	.68	169	.85	.68	855	.35	.71
861	.90	.71	501	.40	.72	162	.90	.72	849	.40	.75
854	.95	.75	494	.45	.76	156	.95	.76	843	.45	.79
.99849	**1.00**	**0.79**	**.99487**	**3.50**	**2.80**	**.99149**	**6.00**	**4.80**	**.98837**	**8.50**	**6.83**
842	.05	.83	480	.55	.84	143	.05	.84	831	.55	.87
834	.10	.87	473	.60	.88	136	.10	.88	825	.60	.91
827	.15	.91	466	.65	.92	130	.15	.92	819	.65	.95
819	.20	.95	459	.70	.96	123	.20	.96	813	.70	.99
812	.25	.99	452	.75	3.00	117	.25	5.00	807	.75	7.03
805	.30	1.03	445	.80	.04	111	.30	.05	801	.80	.07
797	.35	.07	438	.85	.08	104	.35	.09	795	.85	.11
790	.40	.11	431	.90	.12	098	.40	.13	789	.90	.15
782	.45	.15	424	.95	.16	091	.45	.17	783	.95	.19
.99775	**1.50**	**1.19**	**.99417**	**4.00**	**3.20**	**.99085**	**6.50**	**5.21**	**.98777**	**9.00**	**7.23**
768	.55	.23	410	.05	.24	079	.55	.25	771	.05	.27
760	.60	.27	403	.10	.28	072	.60	.29	765	.10	.31
753	.65	.31	397	.15	.32	066	.65	.33	759	.15	.35
745	.70	.35	390	.20	.36	059	.70	.37	754	.20	.39
738	.75	.39	383	.25	.40	053	.75	.41	748	.25	.43
731	.80	.43	376	.30	.44	047	.80	.45	742	.30	.48
723	.85	.47	369	.35	.48	040	.85	.49	736	.35	.52
716	.90	.51	363	.40	.52	034	.90	.53	730	.40	.56
708	.95	.55	356	.45	.56	027	.95	.57	724	.45	.60
.99701	**2.00**	**1.59**	**.99349**	**4.50**	**3.60**	**.99021**	**7.00**	**5.61**	**.98719**	**9.50**	**7.64**
694	.05	.63	342	.55	.64	015	.05	.65	713	.55	.68
687	.10	.67	335	.60	.68	009	.10	.69	707	.60	.72
679	.15	.71	329	.65	.72	002	.15	.73	701	.65	.76
672	.20	.75	322	.70	.76	.98996	.20	.77	695	.70	.80
665	.25	.79	315	.75	.80	990	.25	.81	689	.75	.84
658	.30	.83	308	.80	.84	984	.30	.86	683	.80	.88
651	.35	.87	301	.85	.88	978	.35	.90	678	.85	.92
643	.40	.91	295	.90	.92	971	.40	.94	672	.90	.96
636	.45	.95	288	.95	.96	965	.45	.98	666	.95	8.00

Percentage of alcohol by weight and by volume—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
0.98660	**10.00**	**8.04**	**0.98381**	**12.50**	**10.08**	**0.98114**	**15.00**	**12.13**	**0.97859**	**17.50**	**14.19**
654	.05	.08	375	.55	.12	108	.05	.17	853	.55	.23
649	.10	.12	370	.60	.16	104	.10	.21	848	.60	.27
643	.15	.16	364	.65	.20	099	.15	.25	843	.65	.31
637	.20	.20	359	.70	.24	093	.20	.29	838	.70	.35
632	.25	.24	353	.75	.28	088	.25	.33	833	.75	.40
626	.30	.29	348	.80	.33	083	.30	.38	828	.80	.44
620	.35	.33	342	.85	.37	078	.35	.42	823	.85	.48
614	.40	.37	337	.90	.41	073	.40	.46	818	.90	.52
609	.45	.41	331	.95	.45	068	.45	.50	813	.95	.56
.98603	**10.50**	**8.45**	**.98326**	**13.00**	**10.49**	**.98063**	**15.50**	**12.54**	**.97808**	**18.00**	**14.60**
597	.55	.49	321	.05	.53	057	.55	.58	803	.05	.64
592	.60	.53	315	.10	.57	052	.60	.62	798	.10	.68
586	.65	.57	310	.15	.61	047	.65	.66	793	.15	.73
580	.70	.61	305	.20	.65	042	.70	.70	788	.20	.77
575	.75	.65	299	.25	.69	037	.75	.75	783	.25	.81
569	.80	.70	294	.30	.74	032	.80	.79	778	.30	.85
563	.85	.74	289	.35	.78	026	.85	.83	773	.35	.89
557	.90	.78	283	.40	.82	021	.90	.87	768	.40	.94
552	.95	.82	278	.45	.86	016	.95	.91	763	.45	.98
.98546	**11.00**	**8.86**	**.98273**	**13.50**	**10.90**	**.98011**	**16.00**	**12.95**	**.97758**	**18.50**	**15.02**
540	.05	.90	267	.55	.94	005	.05	.99	753	.55	.06
535	.10	.94	262	.60	.98	001	.10	13.03	748	.60	.10
529	.15	.98	256	.65	11.02	.97996	.15	.08	743	.65	.14
524	.20	9.02	251	.70	.06	991	.20	.12	738	.70	.18
518	.25	.07	246	.75	.11	986	.25	.16	733	.75	.22
513	.30	.11	240	.80	.15	980	.30	.20	728	.80	.27
507	.35	.15	235	.85	.19	975	.35	.24	723	.85	.31
502	.40	.19	230	.90	.23	970	.40	.29	718	.90	.38
496	.45	.23	224	.95	.27	965	.45	.33	713	.95	.39
.98491	**11.50**	**9.27**	**.98219**	**14.00**	**11.31**	**.97960**	**16.50**	**13.37**	**.97708**	**19.00**	**15.43**
485	.55	.31	214	.05	.35	955	.55	.41	703	.05	.47
479	.60	.35	209	.10	.39	950	.60	.45	698	.10	.51
474	.65	.39	203	.15	.43	945	.65	.49	693	.15	.55
468	.70	.43	198	.20	.47	940	.70	.53	688	.20	.59
463	.75	.47	193	.25	.52	935	.75	.57	683	.25	.63
457	.80	.51	188	.30	.56	929	.80	.62	678	.30	.68
452	.85	.55	182	.35	.60	924	.85	.66	673	.35	.72
446	.90	.59	177	.40	.64	919	.90	.70	668	.40	.76
441	.95	.63	172	.45	.68	914	.95	.74	663	.45	.80
.98435	**12.01**	**9.67**	**.98167**	**14.50**	**11.72**	**.97909**	**17.00**	**13.78**	**.97658**	**19.50**	**15.84**
430	.05	.71	161	.55	.76	904	.05	.82	653	.55	.88
424	.10	.75	156	.60	.80	899	.10	.86	648	.60	.93
419	.15	.79	151	.65	.84	894	.15	.90	643	.65	.97
413	.20	.83	146	.70	.88	889	.20	.94	638	.70	16.01
408	.25	.87	140	.75	.93	884	.25	.98	633	.75	.05
402	.30	.92	135	.80	.97	879	.30	14.03	628	.80	.09
397	.35	.96	130	.85	12.01	874	.35	.07	623	.85	.14
391	.40	10.00	125	.90	.05	869	.40	.11	618	.90	.18
886	.45	.04	119	.95	.09	864	.45	.15	613	.95	.22

Percentage of alcohol by weight and by volume—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
0.97608	20.00	16.26	0.97355	22.50	18.34	0.97097	25.00	20.43	0.96828	27.50	22.54
603	.05	.30	350	.55	.38	092	.05	.47	822	.55	.58
598	.10	.34	345	.60	.42	086	.10	.51	816	.60	.62
593	.15	.38	340	.65	.47	081	.15	.56	811	.65	.67
588	.20	.42	335	.70	.51	076	.20	.60	805	.70	.71
583	.25	.46	330	.75	.55	071	.25	.64	800	.75	.75
578	.30	.51	324	.80	.59	065	.30	.68	794	.80	.79
573	.35	.58	319	.85	.63	060	.35	.72	789	.85	.83
568	.40	.59	314	.90	.68	055	.40	.77	783	.90	.88
563	.45	.63	309	.95	.72	049	.45	.81	778	.95	.92
.97558	20.50	16.67	.97304	23.00	18.76	.97044	25.50	20.85	.96772	28.00	22.96
552	.55	.71	299	.05	.80	039	.55	.89	766	.05	23.00
547	.60	.75	294	.10	.84	033	.60	.93	761	.10	.04
542	.65	.80	289	.15	.88	028	.65	.98	755	.15	.09
537	.70	.84	283	.20	.92	023	.70	21.02	749	.20	.13
532	.75	.88	278	.25	.96	018	.75	.06	744	.25	.17
527	.80	.92	273	.30	19.01	012	.80	.10	738	.30	.21
522	.85	.96	268	.35	.05	007	.85	.14	732	.35	.25
517	.90	17.01	263	.40	.09	001	.90	.19	726	.40	.30
512	.95	.05	258	.45	.13	.96996	.95	.23	721	.45	.34
.97507	21.00	17.09	.97253	23.50	19.17	.96991	26.00	21.27	.96715	28.50	23.38
502	.05	.13	247	.55	.21	986	.05	.31	709	.55	.42
497	.10	.17	242	.60	.25	980	.10	.35	704	.60	.47
492	.15	.22	237	.65	.30	975	.15	.40	698	.65	.51
487	.20	.26	232	.70	.34	969	.20	.44	692	.70	.55
482	.25	.30	227	.75	.38	964	.25	.48	687	.75	.60
477	.30	.34	222	.80	.42	959	.30	.52	681	.80	.64
472	.35	.38	216	.85	.46	953	.35	.56	675	.85	.68
467	.40	.43	211	.90	.51	949	.40	.61	669	.90	.72
462	.45	.47	206	.95	.55	942	.45	.65	664	.95	.77
.97457	21.50	17.51	.97201	24.00	19.59	.96937	26.50	21.69	.96658	29.00	23.81
451	.55	.55	196	.05	.63	932	.55	.73	652	.05	.85
446	.60	.59	191	.10	.67	926	.60	.77	646	.10	.89
441	.65	.63	185	.15	.72	921	.65	.82	640	.15	.94
436	.70	.67	180	.20	.76	915	.70	.86	635	.20	.98
431	.75	.71	175	.25	.80	910	.75	.90	629	.25	24.02
426	.80	.76	170	.30	.84	905	.80	.94	623	.30	.06
421	.85	.80	165	.35	.88	899	.85	.98	617	.35	.10
416	.90	.84	159	.40	.93	894	.90	22.03	611	.40	.15
411	.95	.88	154	.45	.97	888	.95	.07	605	.45	.19
.97406	22.00	17.92	.97149	24.50	20.01	.96883	27.00	22.11	.96600	29.50	24.23
401	.05	.96	144	.55	.05	877	.05	.15	594	.55	.27
396	.10	18.00	139	.60	.09	872	.10	.20	587	.60	.32
391	.15	.05	133	.65	.14	866	.15	.24	582	.65	.36
386	.20	.09	128	.70	.18	861	.20	.28	576	.70	.40
381	.25	.13	123	.75	.22	855	.25	.33	570	.75	.45
375	.30	.17	118	.80	.26	850	.30	.37	564	.80	.49
370	.35	.21	113	.85	.30	844	.35	.41	559	.85	.53
365	.40	.26	107	.90	.35	839	.40	.45	553	.90	.57
360	.45	.30	102	.95	.39	833	.45	.50	547	.95	.62

Percentage of alcohol by weight and by volume—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
0.96541	30.00	24.66	0.96235	32.50	26.80	0.95910	35.00	28.96	0.95560	37.50	31.14
535	.05	.70	229	.55	.84	903	.05	29.00	552	.55	.18
529	.10	.74	222	.60	.89	896	.10	.05	545	.60	.23
523	.15	.79	216	.65	.93	889	.15	.09	538	.65	.27
517	.20	.83	210	.70	.97	883	.20	.13	531	.70	.32
511	.25	.87	204	.75	27.02	876	.25	.18	523	.75	.36
505	.30	.91	197	.80	.06	869	.30	.22	516	.80	.40
499	.35	.95	191	.85	.10	862	.35	.26	509	.85	.45
493	.40	25.00	185	.90	.14	855	.40	.30	502	.90	.49
487	.45	.04	178	.95	.19	848	.45	.35	494	.95	.54
.96181	30.50	25.08	.96172	33.00	27.23	.95842	35.50	29.39	.95487	38.00	31.58
475	.55	.12	166	.05	.27	835	.55	.43	480	.05	.63
469	.60	.17	159	.10	.32	828	.60	.48	472	.10	.67
463	.65	.21	153	.15	.36	821	.65	.52	465	.15	.72
457	.70	.25	146	.20	.40	814	.70	.57	457	.20	.76
451	.75	.30	140	.25	.45	807	.75	.61	450	.25	.81
445	.80	.34	133	.30	.49	800	.80	.65	442	.30	.85
439	.85	.38	127	.35	.53	794	.85	.70	435	.35	.90
433	.90	.42	120	.40	.57	787	.90	.74	427	.40	.94
427	.95	.47	114	.45	.62	780	.95	.79	420	.45	.99
.96421	31.00	25.51	.96108	33.50	27.66	.95773	36.00	29.83	.95413	8.50	32.03
415	.05	.55	101	.55	.70	766	.05	.87	405	.55	.07
409	.10	.60	095	.60	.75	759	.10	.92	398	.60	.12
403	.15	.64	088	.65	.79	752	.15	.96	390	.65	.16
396	.20	.68	082	.70	.83	745	.20	30.00	383	.70	.20
390	.25	.73	075	.75	.88	738	.25	.05	375	.75	.25
384	.30	.77	069	.80	.92	731	.30	.09	368	.80	.29
378	.35	.81	062	.85	.96	724	.35	.13	360	.85	.33
372	.40	.85	056	.90	28.00	717	.40	.17	353	.90	.37
366	.45	.90	049	.95	.05	710	.45	.22	345	.95	.42
.96360	31.50	25.94	.96043	34.00	28.09	.95703	36.50	30.26	.95338	39.00	32.46
353	.55	.98	036	.05	.13	695	.55	.30	330	.05	.50
347	.60	26.03	030	.10	.18	688	.60	.35	323	.10	.55
341	.65	.07	023	.15	.22	681	.65	.39	315	.15	.59
335	.70	.11	016	.20	.26	674	.70	.44	307	.20	.64
329	.75	.16	010	.25	.31	667	.75	.48	300	.25	.68
323	.80	.20	003	.30	.35	660	.80	.52	292	.30	.72
316	.85	.24	.95996	.35	.39	653	.85	.57	284	.35	.77
310	.90	.28	990	.40	.43	646	.90	.61	277	.40	.81
304	.95	.33	983	.45	.48	639	.95	.66	269	.45	.86
.96298	32.00	26.37	.95977	34.50	28.52	.95632	37.00	30.70	.95262	39.50	32.90
292	.05	.41	970	.55	.56	625	.05	.74	254	.55	.95
285	.10	.46	963	.60	.61	618	.10	.79	246	.60	.99
279	.15	.50	957	.65	.65	610	.15	.83	239	.65	33.04
273	.20	.54	950	.70	.70	603	.20	.88	231	.70	.08
267	.25	.59	943	.75	.74	596	.25	.92	223	.75	.13
260	.30	.63	937	.80	.78	589	.30	.96	216	.80	.17
254	.35	.67	930	.85	.83	581	.35	31.01	208	.85	.22
248	.40	.71	923	.90	.87	574	.40	.05	200	.90	.27
241	.45	.76	917	.95	.92	567	.45	.10	193	.95	.31

Percentage of alcohol by weight and by volume—Continued.

Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.	Specific gravity at 60° F.	Per cent alcohol by volume.	Per cent alcohol by weight.
0.95185	**40.00**	**33.35**	**0.94786**	**42.50**	**35.58**	**0.94364**	**45.00**	**37.84**	**0.93916**	**47.50**	**40.13**
177	.05	.39	778	.55	.63	355	.05	.89	906	.55	.18
169	.10	.44	770	.60	.67	346	.10	.93	898	.60	.22
161	.15	.48	761	.65	.72	338	.15	.98	888	.65	.27
154	.20	.53	753	.70	.76	329	.20	38.02	879	.70	.32
146	.25	.57	745	.75	.81	320	.25	.07	870	.75	.37
138	.30	.61	737	.80	.85	311	.30	.12	861	.80	.41
130	.35	.66	729	.85	.90	302	.35	.16	852	.85	.46
122	.40	.70	720	.90	.94	294	.40	.21	842	.90	.51
114	.45	.75	712	.95	.99	285	.45	.25	833	.95	.55
.95107	**40.50**	**33.79**	**.94704**	**43.00**	**36.03**	**.94276**	**45.50**	**38.30**	**.93824**	**48.00**	**40.60**
099	.55	.84	696	.05	.08	267	.55	.35	815	.05	.65
091	.60	.88	687	.10	.12	258	.60	.39	805	.10	.69
083	.65	.93	679	.15	.17	250	.65	.44	796	.15	.74
075	.70	.97	670	.20	.21	241	.70	.48	786	.20	.78
067	.75	34.02	662	.25	.23	232	.75	.53	777	.25	.83
059	.80	.06	654	.30	.30	223	.80	.57	768	.30	.88
052	.85	.11	645	.35	.35	214	.85	.62	758	.35	.92
044	.90	.15	637	.40	.39	206	.90	.66	749	.40	.97
036	.95	.20	628	.45	.44	197	.95	.71	739	.45	41.01
.95028	**41.00**	**34.24**	**.94620**	**43.50**	**36.48**	**.94188**	**46.00**	**38.75**	**.93730**	**48.50**	**41.06**
020	.05	.28	612	.55	.53	179	.05	.80	721	.55	.11
012	.10	.33	603	.60	.57	170	.10	.84	711	.60	.15
004	.15	.37	595	.65	.62	161	.15	.89	702	.65	.20
.94996	.20	.42	586	.70	.66	152	.20	.93	692	.70	.24
988	.25	.46	578	.75	.71	143	.25	.98	683	.75	.29
980	.30	.50	570	.80	.75	134	.30	39.03	679	.80	.34
972	.35	.55	561	.85	.80	125	.35	.07	664	.85	.38
964	.40	.59	553	.90	.84	116	.40	.12	655	.90	.43
956	.45	.64	544	.95	.89	107	.45	.16	645	.95	.47
.94948	**41.50**	**34.68**	**.94536**	**44.00**	**36.93**	**.94098**	**46.50**	**39.21**	**.93636**	**49.00**	**41.52**
940	.55	.73	527	.05	.98	089	.55	.26	626	.05	.57
932	.60	.77	519	.10	37.02	080	.60	.30	617	.10	.61
924	.65	.82	510	.15	.07	071	.65	.35	607	.15	.66
916	.70	.86	502	.20	.11	062	.70	.39	598	.20	.71
908	.75	.91	493	.25	.16	053	.75	.44	588	.25	.76
900	.80	.95	484	.30	.21	044	.80	.49	578	.30	.80
892	.85	35.00	476	.35	.25	035	.85	.53	569	.35	.85
884	.90	.04	467	.40	.30	026	.90	.58	559	.40	.90
876	.95	.09	459	.45	.34	017	.95	.62	550	.45	.94
.94868	**42.00**	**35.13**	**.94450**	**44.50**	**37.39**	**.94008**	**47.00**	**39.67**	**.93540**	**49.50**	**41.99**
860	.05	.18	441	.55	.44	.93999	.05	.72	530	.55	42.04
852	.10	.22	433	.60	.48	990	.10	.76	521	.60	.08
843	.15	.27	424	.65	.53	980	.15	.81	511	.65	.13
835	.20	.31	416	.70	.57	971	.20	.85	502	.70	.18
827	.25	.36	407	.75	.62	962	.25	.90	492	.75	.23
810	.30	.40	398	.80	.66	953	.30	.95	482	.80	.27
811	.35	.45	390	.85	.71	944	.35	.99	473	.85	.32
802	.40	.49	381	.90	.76	934	.40	40.04	463	.90	.37
794	.45	.54	373	.95	.80	925	.45	.08	454	.95	.41

3. Determination of Extract.

(a) in distilled liquors, dry wines, beers, ales, etc.

(1) *Direct method.*

Fifty cc are weighed in a platinum dish about 85 mm in diameter and capable of holding about 75 cc and evaporated on the water bath to a sirupy consistence. The residue is heated for two and a half hours in a drying oven at the temperature of boiling water and weighed.

(2) *Optional method.*

Fifty cc of the sample are placed in a platinum or porcelain dish and evaporated on the water bath until the volume is reduced to one-third. The dealcoholized liquid is washed into a 50 cc flask, cooled, and made up to the original volume. It is mixed thoroughly and the specific gravity ascertained with a pycnometer or an accurately standardized hydrometer. The percentage of total solids, in case the pycnometer is used, is obtained from the appended table. The column in the table on the left of the specific gravity gives the percentage of extract in a wine as calculated by Hager, and that on the right the percentage of extract in a beer or wort as calculated by Schultze. According to Baumert, however, Schultze's table gives results which approximate more closely the results obtained by direct estimation than does Hager's.

Tables of Hager and Schultze for the determination of extract by the indirect method.[1]

Hager.	Specific gravity.	Schultze	Hager.	Specific gravity.	Schultze	Hager.	Specific gravity.	Schultze	Hager.	Specific gravity.	Schultze
0.84	1.0038	1.00	1.39	1.0063	1.64	1.94	1.0088	2.28	2.48	1.0113	2.92
0.86	1.0039	1.02	1.42	1.0064	1.67	1.96	1.0089	2.30	2.50	1.0114	2.94
0.88	1.0040	1.05	1.44	1.0065	1.69	1.98	1.0090	2.33	2.52	1.0115	2.97
0.90	1.0041	1.08	1.46	1.0066	1.72	2.00	1.0091	2.35	2.54	1.0116	2.99
0.92	1.0042	1.10	1.48	1.0067	1.74	2.03	1.0092	2.38	2.57	1.0117	3.02
0.94	1.0043	1.13	1.50	1.0068	1.77	2.05	1.0093	2.41	2.59	1.0118	3.05
0.96	1.0044	1.15	1.52	1.0069	1.79	2.07	1.0094	2.43	2.61	1.0119	3.07
0.98	1.0045	1.18	1.55	1.0070	1.82	2.09	1.0095	2.46	2.64	1.0120	3.10
1.00	1.0046	1.21	1.57	1.0071	1.84	2.11	1.0096	2.48	2.66	1.0121	3.12
1.02	1.0047	1.23	1.59	1.0072	1.87	2.14	1.0097	2.51	2.68	1.0122	3.15
1.04	1.0048	1.26	1.61	1.0073	1.90	2.16	1.0098	2.53	2.70	1.0123	3.17
1.06	1.0049	1.29	1.64	1.0074	1.92	2.18	1.0099	2.56	2.72	1.0124	3.20
1.08	1.0050	1.31	1.66	1.0075	1.95	2.21	1.0100	2.58	2.75	1.0125	3.23
1.10	1.0051	1.34	1.68	1.0076	1.97	2.23	1.0101	2.61	2.77	1.0126	3.25
1.12	1.0052	1.36	1.70	1.0077	2.00	2.25	1.0102	2.64	2.79	1.0127	3.28
1.15	1.0053	1.39	1.72	1.0078	2.02	2.27	1.0103	2.66	2.82	1.0128	3.30
1.17	1.0054	1.41	1.75	1.0079	2.05	2.30	1.0104	2.69	2.84	1.0129	3.33
1.19	1.0055	1.44	1.77	1.0080	2.07	2.32	1.0105	2.71	2.86	1.0130	3.35
1.22	1.0056	1.46	1.79	1.0081	2.10	2.34	1.0106	2.74	2.88	1.0131	3.38
1.25	1.0057	1.49	1.82	1.0082	2.12	2.36	1.0107	2.76	2.90	1.0132	3.41
1.27	1.0058	1.51	1.84	1.0083	2.15	2.38	1.0108	2.79	2.92	1.0133	3.43
1.30	1.0059	1.54	1.86	1.0084	2.17	2.40	1.0109	2.82	2.94	1.0134	3.46
1.32	1.0060	1.56	1.88	1.0085	2.20	2.42	1.0110	2.84	2.96	1.0135	3.48
1.34	1.0061	1.59	1.90	1.0086	2.23	2.44	1.0111	2.87	2.98	1.0136	3.51
1.37	1.0062	1.62	1.92	1.0087	2.25	2.46	1.0112	2.89	3.00	1.0137	3.54

[1] If it be desired to use this table for the examination of liquors containing a higher percentage of extract, Schultze's table (intended originally for wort) will be found in Dingler, **230**, 421, and Ztschr. anal. Chem., 1880, **9**, 104, and that of Hager in Ztschr. anal. Chem., 1878, **7**, 503, and Chem. Centlbl., 1878, 415.

(b) IN SWEET WINES.

Ten cc of the liquor are weighed and diluted to 100 cc with water. Fifty cc of this solution are evaporated as described under (1).

4. DETERMINATION OF TOTAL ACIDITY.

Expel any carbon dioxid that is present by continued shaking. Transfer 10 cc to a beaker and, in the case of white wines, add about 10 drops of a neutral litmus solution. Add decinormal sodium hydroxid solution until the red color changes to violet. Then add the reagent, a few drops at a time, until a drop of the liquid, placed on delicate red litmus paper, shows an alkaline reaction. The result is expressed in terms of tartaric acid.

One cc of decinormal sodium hydroxid solution=0.0075 gram tartaric acid.

5. DETERMINATION OF VOLATILE ACIDS.

Fifty cc of wine, to which a little tannin has been added to prevent foaming, are distilled in a current of steam. The flask is heated until the liquid boils, when the lamp under it is turned down, and the steam passed through until 200 cc have been collected in the receiver. The distillate is titrated with decinormal sodium hydroxid solution and the result expressed as acetic acid.

One cc of decinormal sodium hydroxid solution=0.0060 gram acetic acid.

6. DETERMINATION OF GLYCEROL.

(a) IN DRY WINES.

One hundred cc of wine are evaporated in a porcelain dish to about 10 cc, a little quartz sand and milk of lime added, and the evaporation carried almost to dryness. The residue is mixed with 50 cc of 90 per cent alcohol, using a glass pestle or spatula to break up any solid particles, heated to boiling on the water bath, allowed to settle, and the liquid filtered into a small flask. The residue is repeatedly extracted in a similar manner with small portions of boiling alcohol until the filtrate in the flask amounts to about 150 cc. A little quartz sand is then added to it, the flask connected with a condenser, and the alcohol slowly distilled until about 10 cc remain. The evaporation is then continued on the water bath until the residue becomes sirupy. It is cooled and dissolved in 10 cc of absolute alcohol. The solution may be facilitated by gentle heating on the water bath. Fifteen cc of anhydrous ether are added and the flask stoppered and allowed to stand until the precipitate has collected on the sides and bottom of the flask. The clear liquid is decanted into a tared weighing bottle, the precipitate repeatedly washed with a few cubic centimeters of a mixture of one part absolute alcohol and 1.5 parts anhydrous ether and the washings added to the solution. The ether-alcohol is evaporated on the water bath and the residue dried one hour in a water oven, weighed, the amount of ash determined, and its weight deducted from that of the weighed residue.

(b) IN SWEET WINES.

One hundred cc of wine are evaporated on the water bath to a sirupy consistence a little quartz sand being added to render subsequent extraction easier. The residue is repeatedly extracted with absolute alcohol until the united extracts amount to from 100 to 150 cc. The extract is collected in a flask and for every part of alcohol 1.5 parts of ether are added, the liquid well shaken, and allowed to stand until it becomes clear. The supernatant liquid is decanted into a beaker, and the precipitate washed with a few cubic centimeters of a mixture of one part alcohol and 1.5 parts ether. The united liquids are distilled, the evaporation being finished on the water bath, the residue is dissolved in water, transferred to a porcelain dish, and treated as under (a).

It is necessary to test the glycerol from sweet wines for sugar, and if any is present it must be estimated by Fehling-Soxhlet's method, page 50, and its weight subtracted from that of the glycerol.

7. Determination of Reducing Sugars.

The reducing sugars are estimated as dextrose, and may be determined by any of the methods given for the estimation of dextrose, under Analysis of Carbohydrates (p. 53).

8. Polarization.

All results are to be stated as the polarization of the undiluted wine. The Schmidt and Haensch half-shadow saccharimeter is to be used, and the results expressed in terms of the sugar scale of this instrument. If any other instrument be used, or if it be desirable to convert to angular rotation, the following factors may be employed:

1° Schmidt and Haensch = 0.3468° angular rotation D.
1° angular rotation D = 2.8835° Schmidt and Haensch.
1° Schmidt and Haensch = 2.6048° Wild (sugar scale).
1° Wild (sugar scale) = 0.3840° Schmidt and Haensch.
1° Wild (sugar scale) = 0.1331° angular rotation D.
1° angular rotation D = 0.7511° Wild (sugar scale).
1° Laurent (sugar scale) = 0.2167° angular rotation D.
1° angular rotation D = 4.6154° Laurent (sugar scale).

(a) in white wines.

Sixty cc of wine are decolorized with 3 cc of lead subacetate solution and filtered. Thirty cc of the filtrate are treated with 1.5 cc of a saturated solution of sodium carbonate, filtered, and polarized. This gives a dilution of 10 to 11, which must be considered in the calculation, and the polariscope reading must accordingly be increased one-tenth.

(b) in red wines.

Sixty cc of wine are decolorized with 6 cc of lead subacetate solution and filtered. To 30 cc of the filtrate 3 cc of a saturated solution of sodium carbonate are added, filtered, and the filtrate polarized. The dilution in this case is 5 to 6, and the polariscope reading must accordingly be increased one-fifth.

(c) in sweet wines.

(1) *Before inversion.*

One hundred cc are decolorized with 2 cc of lead subacetate solution and filtered after the addition of 8 cc of water. One-half cc of a saturated solution of sodium carbonate and 4.5 cc. of water are added to 55 cc. of the filtrate, and the liquid mixed, filtered, and polarized. The polariscope reading is multiplied by 1.2.

(2) *After inversion.*

Thirty-three cc of the filtrate from the lead subacetate in (1) are placed in a flask with 3 cc strong hydrochloric acid. After mixing well the flask is placed in water and heated until a thermometer, placed in the flask with the bulb as near the center of the liquid as possible, marks 68°, consuming about fifteen minutes in the heating. It is then removed, cooled quickly to room temperature, filtered, and polarized, the temperature being noted. The polariscope reading is multiplied by 1.2.

(3) *After fermentation.*

Fifty cc of wine, which have been dealcoholized and made up to the original volume with water, are mixed in a small flask with well-washed beer yeast and kept at 30° until fermentation has ceased, which requires from two to three days. The liquid

is then washed into a 100 cc flask, a few drops of a solution of acid mercuric nitrate and then lead-subacetate solution, followed by sodium carbonate, added. The flask is filled to the mark with water, shaken, and the solution filtered and polarized.

(d) APPLICATION OF ANALYTICAL METHODS.

(1) *The wine shows no rotation.*

This may be due to the absence of any rotatory body, to the simultaneous presence of the dextrorotatory nonfermentable constituents of commercial dextrose and levorotatory sugar, or to the simultaneous presence of dextrorotatory cane sugar and levorotatory invert sugar.

(*a*) THE WINE IS INVERTED.—A levorotation shows that the sample contained cane sugar.

(*b*) THE WINE IS FERMENTED.—A dextrorotation shows that both levorotatory sugar and the unfermentable constituents of commercial dextrose were present.

If no change takes place in either (*a*) or (*b*) in the rotation it proves the absence of unfermented cane sugar, the unfermentable constituents of commercial dextrose, and of levorotatory sugar.

(2) *The wine rotates to the right.*

This may be caused by unfermented cane sugar, the unfermentable constituents of commercial dextrose, or both.

(*a*) THE WINE IS INVERTED.

(a_1) *It rotates to the left after inversion.*—Unfermented cane sugar was present.

(a_2) *It rotates more than 2.3° to the right.*—The unfermentable constituents of commercial dextrose are present.

(a_3) *It rotates less than 2.3° and more than 0.9° to the right.*—It is in this case treated as follows:

Two hundred and ten cc of the wine are evaporated in a porcelain dish to a thin sirup with a few drops of a 20 per cent solution of potassium acetate. To the residue 200 cc of 90 per cent alcohol are added with constant stirring. The alcoholic solution is filtered into a flask, and the alcohol removed by distillation until about 5 cc remain. The residue is mixed with washed bone black, filtered into a graduated cylinder, and washed until the filtrate amounts to 30 cc. When the filtrate shows a dextrorotation of more than 1.5°, it indicates the presence of the unfermentable constituents of commercial dextrose.

(3) *The wine rotates to the left.*

It contains unfermented levorotatory sugar, derived either from the must or from the inversion of added cane sugar. It may, however, also contain unfermented cane sugar and the unfermentable constituents of commercial dextrose.

(*a*) The wine is fermented according to 8, (c), (3).

(a_1) It polarizes 3° after fermentation. It contains only levorotatory sugar.

(a_2) It rotates to the right. It contains both levorotatory sugar and the unfermentable constituents of commercial dextrose.

(*b*) The wine is inverted according to 8, (c), (2).

(b_1) It is more strongly levorotatory after inversion. It contains both levorotatory sugar and unfermented cane sugar.

9. Determination of Tannin and Coloring Matter.

(a) preparation of reagents.

Decinormal solution of oxalic acid.—10 cc=0.04157 gram tannin.

Potassium permanganate solution.—1.333 grams of potassium permanganate are dissolved in 1 liter of water and the solution standardized by means of the decinormal oxalic acid solution.

Indigo solution.—Six grams of sodium indigo sulfate (sodium sulfindigotate) are dissolved in 500 cc of water with the aid of heat, cooled, 50 cc of concentrated sulfuric acid added, and the solution made up to 1 liter, and filtered.

Purified bone black.—Finely pulverized bone black is extracted with hydrochloric acid and washed with distilled water until the acid is entirely removed. The bone black is kept covered with water.

(b) determination.

(1) One hundred cc of wine are dealcoholized by evaporation, and the original volume restored with water. Ten cc are measured into a porcelain casserole having a capacity of about a liter, and 750 cc of water and 20 cc of the indigo solution added, the latter being measured from a burette. The potassium permanganate solution is added, a cubic centimeter at a time, until the blue color changes to green, then a few drops at a time until the liquid becomes golden yellow. Designate by *a* the number of cubic centimeters of permanganate solution used.

(2) Ten cc of the dealcoholized wine, obtained as in (1), are treated with bone black for fifteen minutes, filtered, and the bone black washed carefully. The filtrate is diluted with 750 cc of water, 20 cc of indigo solution added, and the titration carried out as in (1). Designate burette reading by *b*.

Then $a-b=c$=the number of cubic centimeters of the potassium permanganate solution required for the oxidation of the tannin and coloring matter in 10 cc of wine.

10. Determination of Potassium Bitartrate.

The determination of potassium bitartrate is necessary when an estimation of the tartaric acid is desired.

Fifty cc of wine are placed in a porcelain dish and evaporated to a sirupy consistence, a little quartz sand being added to render subsequent extraction easier. After cooling, 70 cc of 96 per cent alcohol are added with constant stirring. After standing for twelve hours at as low a temperature as practicable, the solution is filtered and the precipitate washed with alcohol until the filtrate is no longer acid. The alcoholic filtrate is preserved for the estimation of the tartaric acid. The filter and precipitate are returned to the porcelain dish and repeatedly treated with hot water, each extraction being filtered into a flask or beaker until the washings are neutral. The combined aqueous filtrates and washings are titrated with decinormal sodium hydroxid solution.

One cc decinormal sodium hydroxid solution=0.0188 gram potassium bitartrate.

11. Determination of Tartaric Acid.

(a) in the alcoholic filtrate from the potassium bitartrate 10.

The filtrate is made up to a definite volume with water and divided into two equal portions. One portion is exactly neutralized with decinormal sodium hydroxid solution, the other portion added, the alcohol evaporated, the residue washed into a porcelain dish, and treated as under 10.

One cc decinormal sodium hydroxid solution=0.0075 gram tartaric acid.

As, however, only half of the free tartaric acid is determined by this method—

One cc decinormal sodium hydroxid =0.0150 gram of tartaric acid.

(b) MODIFIED BERTHELOT-FLEURY METHOD.

Ten cc of wine are neutralized with potassium hydroxid solution and mixed in a graduated cylinder with 40 cc of the same sample. To one-fifth of the volume, corresponding to 10 cc of wine, 50 cc of a mixture of equal parts of alcohol and ether are added and allowed to stand twenty-four hours. The precipitated potassium bitartrate is separated by filtration, dissolved in water, and titrated. The excess of potassium bitartrate over the amount of that constituent present in the wine corresponds to the free tartaric acid.

12. DETERMINATION OF TARTARIC, MALIC, AND SUCCINIC ACIDS.

[Schmidt and Hiepe's method.]

Two hundred cc of wine are evaporated one-half, cooled, and lead subacetate solution added until the reaction is alkaline. The precipitate is separated by filtration and washed with cold water until the filtrate shows only a slight reaction for lead. The precipitate is washed from the filter into a beaker by means of hot water, and treated hot with hydrogen sulfid until all the lead is converted into sulfid. It is then filtered hot and the lead sulfid washed with hot water until the washings are no longer acid. The filtrate and washings are evaporated to 50 cc and accurately neutralized with potassium hydroxid. An excess of a saturated solution of calcium acetate is added and the liquid allowed to stand from four to six hours with frequent stirring. It is then filtered and the precipitate washed until the filtrate amounts to exactly 100 cc. The precipitate of calcium tartrate is converted into calcium oxid by igniting in a platinum crucible. After cooling, from 10 to 15 cc of normal hydrochloric acid are added, the solution washed into a beaker, and accurately titrated with normal potassium hydroxid solution. Every cubic centimeter of normal acid saturated by the calcium oxid is equivalent to 0.0750 gram tartaric acid. To the amount so obtained, 0.0286 gram must be added, representing the tartaric acid held in solution in the filtrate as calcium tartrate. The sum represents the total tartaric acid in the wine.

The filtrate from the calcium tartrate is now evaporated to about 25 cc, cooled, and mixed with three times its volume of 96 per cent alcohol. After standing several hours the precipitate is collected on a weighed filter, dried at 100°, and weighed. It represents the calcium salts of malic, succinic, and sulfuric acids and of the tartaric acid which remained in solution. This precipitate is dissolved in a minimum quantity of hydrochloric acid, filtered, and the filter washed with hot water. Potassium carbonate solution is added to the hot filtrate and the precipitated calcium carbonate separated by filtration and washed. This filtrate contains the potassium salts of the above-named acids. It is neutralized with acetic acid, evaporated to a small volume, and precipitated hot with barium chlorid. The precipitate of barium succinate and sulfate is separated by filtration, washed with hot water, and treated on the filter with dilute hydrochloric acid. The barium sulfate remaining is washed, dried, ignited, and weighed. In the filtrate which contains the barium succinate, the barium is precipitated hot with sulfuric acid, washed, dried, ignited, and weighed. Two hundred and twenty-three parts barium sulfate equal 118 parts succinic acid. The succinic and sulfuric acids, as well as the tartaric acid remaining in solution which was equal to 0.0286, are to be calculated as calcium salts and the result deducted from the total weight of the calcium precipitate. The remainder is the calcium malate, of which 172 parts equal 134 parts malic acid.

13. Detection of Coloring Matter.

(a) cazeneuve reaction.

Add 0.2 gram of precipitated mercuric oxid to 10 cc of wine, shake for one minute, and filter.

Pure wines give filtrates which are colorless or light yellow, while the presence of a more or less red coloration indicates that an anilin color has been added to the wine.

(b) method of sostegni and carpentieri.

Evaporate the alcohol from 200 cc of wine, add 2 to 4 cc of a 10 per cent solution of hydrochloric acid, immerse some threads of fat-free wool, and boil for five minutes. Remove the threads, wash them with cold water acidified with hydrochloric acid, then with hot water acidified with hydrochloric acid, then with pure water, and dissolve the color in a boiling mixture of 50 cc of water and 2 cc of concentrated ammonia. Replace the threads by new ones, acidify with hydrochloric acid, and boil again for five minutes. In the presence of anilin colors to the amount of 2 mg per liter, the threads are dyed as follows:

Safranin	light rose-red.
Vinolin	rose-red to violet.
Bordeaux red	rose-red to violet.
Ponceau red	rose-red.
Fuchsin	dirty white.
Tropæolin 00	straw yellow.
Tropæolin 000	light orange.
Corallin	dirty white.

This method is not suitable for the detection of fuchsin or corallin.

(c) detection of fuchsin and orseille.

To 20 cc of wine add 10 cc of lead acetate solution, heat slightly, and mix by shaking. Filter into a test tube, add 2 cc of amyl alcohol, and shake. If the amyl alcohol be colored red, separate it and divide it into two portions. To one add hydrochloric acid, to the other ammonia. When the color is due to fuchsin, the amyl alcohol will in both cases be decolorized; when due to orseille, the ammonia will change the color of the amyl alcohol to purple-violet.

14. Determination of Ash.

The residue from the direct extract determination is incinerated at as low a heat as possible. Repeated moistening, drying, and heating to redness is advisable to get rid of all organic substances.

15. Determination of Potash.

(a) kayser's method.

Dissolve 0.7 gram of pure sodium hydroxid and 2 grams of tartaric acid in 100 cc of wine, add 150 cc of 92–94 per cent alcohol, and allow the liquid to stand twenty-four hours. The precipitated potassium bitartrate is collected on a small filter and washed with 50 per cent alcohol until the filtrate amounts to 260 cc. The precipitate and filter are transferred to the beaker in which the precipitation was made, the precipitate dissolved in hot water, the volume made up to 200 cc, and 50 cc titrated with decinormal sodium hydroxid solution; 0.004 gram must be added to the final result, this representing the potash which remains in solution as bitartrate.

(b) PLATINUM CHLORID METHOD.

Evaporate 100 cc of wine to dryness, incinerate the residue, and determine potash as given under Methods for the Analysis of Fertilizers (p. 22).

16. DETERMINATION OF SULFUROUS ACID.

One hundred cc of wine are distilled in a current of carbon dioxid after the addition of phosphoric acid until about 50 cc have passed over. The distillate is collected in accurately standardized iodin solution. When the distillation is finished, the excess of iodin is determined with standardized sodium thiosulfate solution, and the sulfurous acid calculated from the iodin used.

17. DETECTION OF SALICYLIC ACID.

(a) SPICA'S METHOD.

Acidify 100 cc of the liquor with sulfuric acid and extract with sulfuric ether. Evaporate the extract to dryness, warm the residue carefully with 1 drop of concentrated nitric acid, and add 2 or 3 drops of ammonia. The presence of salicylic acid in the liquor is indicated by the formation of the yellow color of ammonium picrate, and may be confirmed by dyeing a thread of fat-free wool.

(b) BIGELOW'S METHOD.

Place 100 cc of the wine in a separatory funnel, add 5 cc of sulfuric acid (1-3), and extract with a sufficient quantity of a mixture of eight or nine parts of ether to one part of petroleum ether. Throw away the aqueous solution, wash the ether once with water, then shake thoroughly with about 50 cc of water, to which from 6 to 8 drops of 0.5 per cent solution of ferric chlorid have been added. Discard the aqueous solution, which contains the greater part of the tannin in combination with iron, wash again with water, transfer the ethereal solution to a porcelain dish, and allow to evaporate spontaneously. Then heat the dish on the steam bath, take up with 1 or 2 cc of cold water, filter into a test tube, and add 1 or 2 drops of 0.5 per cent solution of ferric chlorid. The presence of salicylic acid is indicated by the appearance of a violet-red coloration. In the case of red wines a second extraction of the residue with ether mixture is sometimes necessary. This is determined by the amount of residue left in the dish on the evaporation of the ether.

This method can not be used in the examination of beers and ales.

(c) GIRARD'S METHOD.

Extract a portion of the acidified liquor with ether as in the preceding methods, evaporate the extract to dryness, and extract the residue with petroleum ether. The residue from the petroleum ether extract is dissolved in water and treated with a few drops of a very dilute solution of ferric chlorid. The presence of salicylic acid is indicated by the appearance of a violet-red coloration.

18. DETECTION OF GUM AND DEXTRIN.

Four cc of wine are mixed with 10 cc of 96 per cent alcohol. When gum arabic or dextrin is present, a lumpy, thick, and stringy precipitate is produced, whereas pure wine becomes at first opalescent and then gives a flocculent precipitate.

VIII.—METHODS FOR THE ANALYSIS OF TANNING MATERIALS.

1. PREPARATION OF SAMPLE.

Barks, woods, leaves, and similar tanning materials should be ground to such a degree of fineness that they can be thoroughly extracted.

2. Testing the Hide Powder.

Shake 10 grams of the hide powder with 200 cc of water for five minutes; filter through muslin or linen; squeeze out thoroughly by hand; collect the hide powder, and repeat the operation twice with the same quantity of water. Pass the last filtrate through paper until a perfectly clear liquid is obtained. Evaporate 100 cc of the final filtrate in a weighed dish; dry at 100° until the weight is constant. If the residue amount to more than 10 mg the sample should be rejected. The hide powder must be kept in a dry place and tested once a month.

3. Determination of Moisture.

Dry 2 grams of the sample in a flat-bottomed dish at the temperature of boiling water until the weight becomes constant.

4. Quantity of Tanning Material.

Use such an amount of the tanning material as shall give in 100 cc of the filtered solution about 1 gram of dry solids. In the case of barks, woods, leaves, and similar materials transfer to a 500 cc flask; fill the flask with water at approximately 80° and let stand overnight in a bath which is kept at 80°; cool, make up to the mark, shake well, and filter. In the case of extracts and sweet liquors, wash into a 500 cc flask with water at approximately 80°, almost filling the flask, cool, and make up to mark.

5. Determination of Total Solids.

[Applicable only to extracts and sweet liquors.]

Shake the solution and immediately remove 100 cc with a pipette, evaporate in a weighed dish, and dry to constant weight at the temperature of boiling water. The dishes in which the drying is conducted should be covered while they are in the oven, in order to prevent loss.

6. Determination of Soluble Solids.

Filter the remainder of the solution through a folded filter, adding a teaspoonful of kaolin, if necessary. Evaporate 100 cc of the filtrate and dry as under 5.

7. Determination of Tanning Substances.

Prepare 10 grams of hide powder as indicated under 1, filter through well washed muslin or linen, and dry as much as possible in a suitable press. Weigh the wet powder and determine the residual moisture in about 3 grams by drying to constant weight at 60°. Divide the remaining powder into three portions. Shake 150 cc of the filtered solution obtained as directed under 6 with one portion of moist hide powder for about five minutes, and filter through muslin or linen. To the filtered liquor add the second portion of hide powder and shake again for about five minutes. Filter as before, add the third portion of hide powder, and shake for about eight minutes. Pass through filter paper, pouring the filtrate back until it runs through clear. Evaporate 100 cc of the clear filtrate and dry the residue to constant weight at the temperature of boiling water. The difference between the soluble solids obtained as directed in 6 and the last residue is the amount of tanning material absorbed by the hide powder. This must be corrected for the water retained by the hide powder. The shaking must be conducted by means of a mechanical shaker, as shaking by hand is not intense enough to remove all the tanning substances from the solution. The simple machine used by druggists, and known as the milk-shake, is recommended.

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